LABORATORY MANUAL
OF
INORGANIC AND ORGANIC
PHARMACEUTICAL PREPARATIONS

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LABORATORY MANUAL

OF

Inorganic and Organic Pharmaceutical Preparations

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WITH NOTES AND EXPLANATIONS BASED UPON EXTENDED EXPERIENCE

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PREFACE

Practice in the Manufacture of Pharmaceutical Preparations is now recognized as necessary to the proper education of pharmacists. In no other occupation is the application of modern educational methods more important. The notes and explanations presented in the text enable the student to do his work intelligently and successfully, and he should have his Manual before him on the work table.

The lessons have been selected with due regard to practical illustrations of the didactic courses, and the ample variety included will permit a wide choice. The methods laid down have been thoroughly tested and we believe them to be as nearly faultless as diligent attention during twenty-five years can make them.

THE AUTHORS.

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School of Pharmacy,
Chicago, 1911.

ACIDS.

ACETIC ACID.

$HC_2H_3O_2=60$.

Sodium acetate	18 parts
Sulphuric acid	13 parts

Reduce the acetate to a coarse powder; introduce it into a distilling flask; warm the flask; connect it with a condenser and receiver; add the sulphuric acid through a funnel tube, a little at a time, and continue the distillation until 12 parts of distillate have been collected.

Test the product with volumetric test-solution of potassium hydroxide to determine its strength and then dilute as required.

Properties. A solution composed of 36 per cent of hydrogen acetate and 64 per cent of water.

A clear, colorless liquid, of a distinctly vinegar-like odor, a purely acid taste, and a strongly acid reaction. Sp. gr. 1.048 at 15°C., compared with water at +4° C. as 1.000. Its specific volume is accordingly 0.954. Miscible in all proportions with water and alcohol, and wholly volatilized by heat.

The presence of empyreuma is at once detected by a smoky odor when the acid is neutralized with KOH, NaOH, or $KHCO_3$.

An acetic acid of from 77 to 80 per cent strength has the sp. w. of about 1.0748; weaker and stronger solutions of acetic acid have a lower specific weight. Hence the strength of acetic acid can not be determined otherwise than by actual assay; unless the acid is diluted with an equal volume of water and the sp. w. of the mixture taken; then, from a table, the percentage of acid indicated by the sp. w. is ascertained, and multiplied by two.

BORIC ACID.

$H_3BO_3=62$.

Borax, in powder	100 parts
Nitric acid	54 parts
Distilled water.	

Dissolve the borax in 250 parts of boiling distilled water, and filter while hot. Add the acid to the hot filtrate and stir. Set aside in a cold place for a day.

Collect the crystals on a muslin or paper filter and wash them with a small quantity of cold distilled water.

Re-dissolve the washed crystals in 200 parts of boiling distilled water, and set the solution aside until quite cold.

Place a loose layer of clean cotton in the throat of a glass funnel and collect the crystals upon it. Drain thoroughly. Transfer the product to a dry muslin cloth or a sheet of bibulous white paper, spread it out, and dry it in a moderately warm place.

Reaction. $Na_2B_4O_7.10H_2O+2HNO_3=4H_3BO_3$
$+2NaNO_3+5H_2O$.

Notes. A slight excess of acid is necessary. Nitric acid is preferred to hydrochloric acid because sodium nitrate is more readily soluble than sodium chloride. Sulphuric acid is not employed because it is not so readily washed out.

The yield is theoretically about two-thirds of the weight of the borax used, but only about 50 to 60 per cent can be obtained in practice.

Description.—Colorless or white, pearly, lamellar crystals, somewhat unctuous to the touch; taste feebly acid, slightly bitterish. Boric acid dissolves in 30 parts of water at 15° C.;

in 26 parts at 19°; 15 parts at 25°; about 4.75 parts at 75°; 3.55 parts at 87.5°; and in 2.97 parts of boiling water.

Or, 100 parts of water dissolves 3.9 parts of boric acid at 19°; 6.8 parts at 25°; 9.8 parts at 50°; 21 parts at 75°; 28 parts at 87.5°; and 34 parts at 100°.

DILUTED HYDROBROMIC ACID.

$HBr=80$.

Sulphuric acid	14 parts
Potassium bromide	12 parts
Distilled water, sufficient.	

Put two parts of the water in a porcelain dish capable of holding about 32 parts; stir the water into a rapid rotary motion; then add the sulphuric acid slowly and in a thin stream, continuing the stirring with a glass rod.

Put the potassium bromide, previously reduced to coarse powder, in another dish; add 13 parts of water, heat the mixture by a sand-bath until the salt is dissolved, and while the solution is still hot add to it the sulphuric acid mixture, stirring briskly. Set aside for twenty-four hours.

Decant the acid supernatant liquid (hydrobromic acid) from the crystals of potassium sulphate, and pour it into a suitable distilling flask. Wash the crystals with a little cold water, and add these washings to the liquid.

Provide the flask with a perforated stopper and safety tube; connect it with a well cooled receiver, and distill nearly to dryness. Assay the distillate and dilute it with enough distilled water to make the final product contain 10 per cent of hydrogen bromide.

Reaction. $2KBr+H_2SO_4=K_2SO_4+2HBr$.

Description.—A clear, colorless, odorless, strongly acid liquid having a sp. w. of about 1.077 at 15°.

It is to be kept in glass-stoppered bottles in a cool, dark place.

PURIFIED OXALIC ACID.

$H_2C_2O_4.2H_2O=126$.

Commercial oxalic acid	1000 parts
Hydrochloric acid (31.9%HCl)......	300 parts
Water.	

Put the oxalic acid in a porcelain dish and add 700 parts of hot water. Boil for a few minutes. Let it settle. Decant the solution while still hot from the undissolved portion (which consists mostly of oxalates and may be reserved for further treatment, if profitable). Add the hydrochloric acid to the hot solution obtained. Stir well. Cool rapidly, stirring the liquid to get small crystals. Collect the crystals and wash the product with a very small quantity of cold water.

Recrystallize the product once or twice as may be required.

Notes. The less soluble oxalates remain undissolved when commercial oxalic acid is treated with less water than is required for complete solution. The remaining oxalates are decomposed by the hydrochloric acid, which is used in considerable excess. It is best not to allow the oxalic acid to form large crystals from the solution containing hydrochloric acid; small crystals are more readily washed free from the HCl. Recrystallization from a hot saturated solution is necessary to complete the purification, and it may be found necessary to repeat it.

Description.—Colorless or white prisms, readily soluble in water and alcohol. It has a strong acid taste, and is a powerful poison in large doses because so corrosive.

Pure oxalic acid volatilizes on platinum without residue.

OTHER PREPARATIONS.

ALUM.

(*Potassium Alum. Aluminum-Potassium Sulphate.*)

$AlK(SO_4)_2.12H_2O=474.$

Aluminum sulphate	25 parts
Potassium sulphate	7 parts
Water	60 parts
Diluted sulphuric acid (10%)	1 part

Heat the water to boiling. Add the aluminum sulphate. Then add the potassium sulphate, stirring well. Add the diluted sulphuric acid. Filter if necessary. Set the solution aside to cool and crystallize.

Evaporate the mother-liquid to obtain additional crops of crystals as far as practicable. Recrystallize the product once or twice as may be necessary.

Crystallized Alum.

Potash alum	2 parts
Water	18 parts
Diluted sulphuric acid	1 part

Make a solution with the aid of heat. Filter. Crystallize the salt in the usual way.

Very large and well-developed crystals may be easily obtained if the crystallization proceeds slowly by spontaneous evaporation and with the solution at perfect rest. To start crystallization a few small crystals of pure potash alum may be placed in the solution. The crystals should not be permitted to rest on one side throughout the process, but should be turned over occasionally in order that they may be equally well developed on all sides.

Turbidated Alum.

Potash alum	20 parts
Boiling water	30 parts
Diluted sulphuric acid	1 part

Dissolve, filter, and cool the solution rapidly, stirring it constantly, to as low a temperature above the freezing point of water as may be conveniently attained. Collect, drain and dry the crystalline salt.

Description.—Alum consists of large, colorless, octohedral crystals, sometimes modified by cubes, or occurs in crystalline fragments, without odor, but having a sweetish and strongly astringent taste. On exposure to the air, the crystals are liable to absorb ammonia, and acquire a whitish coating.

Soluble in 9 parts of water at 15° C., and in 0.3 part of boiling water; it is also freely soluble in warm glycerin, but is insoluble in alcohol.

The salt has an acid reaction upon litmus paper.

ALUMINUM HYDROXIDE.

$Al(OH)_3=78.$

Alum, in powder	1 part
Sodium carbonate	1 part
Distilled water, sufficient.	

Dissolve each salt in 15 parts of distilled water, filter the solutions and heat to boiling. Then having poured the hot solution of carbonate of sodium into a capacious vessel, gradually pour into it the hot solution of alum with constant stirring, and add about 10 parts of boiling distilled water. Let the precipitate subside, decant the clear liquid, and pour upon the precipitate 20 parts of hot distilled water. Again decant, transfer the precipitate to a strainer, and wash it with hot distilled water until the washings give but a faint cloudiness with test-solution of barium chloride. Then allow it to drain, dry it at a temperature not exceeding 40° C., and reduce it to a uniform powder.

Reaction.

$$3Na_2CO_3+2AlK(SO_4)_2+3H_2O$$
$$=3Na_2SO_4+K_2SO_4+2Al(OH)_3+3CO_2.$$

Notes. It is necessary to have a decided excess of alkali present throughout the process, and as equal parts of alum and sodium carbonate leave only about ten per cent excess of the alkali, and as this excess is not too great, these proportions are used. Unless a sufficient excess of alkali be present from the beginning to the end of the reaction, and the liquid still remains alkaline after all the alum has been decomposed, the product will contain sulphate. This explains why it is directed that the alum solution must be poured into the alkali solution, and not *vice versa;* it also accounts for the injunction to add the alum solution slowly and with constant stirring.

Aluminum hydroxide is a very light, voluminous precipitate, forming an almost gelatinous magma, which settles very slowly if at all, and is therefore difficult to wash. It will of course settle less readily in a dense liquid than in one less dense. Hence the addition of hot water greatly facilitates the washing by decantation. But prolonged contact with boiling water changes the constitution of the hydroxide from $Al(OH)_3$ to $OAlOH$, and the latter compound is insoluble in acids. Hence, the liquid in which the hydroxide is suspended must not be boiling, but only hot.

The washed hydroxide is to be dried without much heat in order to avoid its becoming hard and gritty.

It is chiefly prepared as a preliminary step in the preparation of sulphate, chloride, nitrate, acetate, and other aluminum salts. When prepared for this purpose the hydroxide, after having been well washed, is simply drained but not dried before dissolved in the proper acid.

Description.—A white, light, amorphous, odorless and tasteless powder, insoluble in water and in alcohol.

ALUMINUM OLEATE.

$Al(C_{18}H_{33}O_2)_3=870.$

Potash alum	60 Gm
White Castile soap, in fine powder	110 Gm

Dissolve the alum in 3,600 ml of water, and the soap in 1,800 ml of hot water. Pour the cold soap solution gradually into the alum solution. Warm the mixture until the oleate separates. Decant the mother liquor, wash the oleate twice with hot water, using 3,000 ml of water each time, and then collect the product.

Reaction.

$$6NaC_{18}H_{33}O_2+2KAl(SO_4)_2 =2Al(C_{18}H_{33}O_2)_3+K_2SO_4+3Na_2SO_4.$$

Notes. To free this oleate from water it must be melted in an evaporating dish by very gentle heat over the water-bath. The yield is about 100 Gm or slightly over that amount.

Description.—Aluminum oleate is a soft, white plaster, containing 5.86 per cent of aluminum oxide.

PURE ALUMINUM SULPHATE.

$Al_2(SO_4)_3.16H_2O=630.$

Alum	30 parts
Sodium carbonate	30 parts
Sulphuric acid	9 parts

Dissolve the alum and the sodium carbonate, each in 400 parts of boiling water, filter, and pour the hot alum solution into the hot solution of sodium carbonate, stirring constantly. Digest the mixture at a gentle heat until the evolution of carbon dioxide ceases. Wash the precipitate with hot water until the washings no longer yield a precipitate with test solution of barium chloride. Then let the precipitated aluminum hydrate drain as far as possible, and dissolve it with the aid of gentle heat in the sulphuric acid, previously diluted with 50 parts of water; filter the solution, evaporate it with constant stirring, over a water bath, until a nearly dry white salt remains.

Notes. Aluminic hydroxide is first prepared. See article Aluminum Hydroxide, for reaction and notes. Then the sulphuric acid is saturated with the aluminum hydroxide. Care must be taken not to lose any of the hydroxide in the process of washing it, for if the quantity is insufficient the acid can not be saturated and the product will then contain free sulphuric acid. If there is any reason for believing that any portion of the aluminum hydroxide has been wasted, the amount of sulphuric acid used may be reduced so as to insure its saturation.

Description.—Pure aluminum sulphate is a white crystalline powder; odorless; taste sweetish, astringent. Permanent in the air if properly prepared. Soluble in 1.2 parts of water at 15°. Insoluble in alcohol.

AMMONIUM BROMIDE.

$H_4NBr=97$.

Iron wire	4 parts
Bromine	12 parts
Ammonia water	25 parts
Distilled water.	

Put the iron wire into a flask, add 50 parts of warm (not *hot*) water, and then gradually add 9 parts of bromine. Put a loose plug of cotton into the neck of the flask, and shake gently from time to time until a greenish liquid is obtained having no odor of bromine. Filter this solution of ferrous bromide, add the remainder of the bromine to the filtrate and shake gently so that a uniform solution may be obtained.

Pour this solution into the ammonia water previously diluted with 50 parts of distilled water. Shake well. Heat the mixture over the water-bath for half an hour. Then filter. When the liquid has passed through the filter, wash the residue upon it with some hot distilled water, letting the washings run into the filtered solution.

Evaporate the liquid in a porcelain dish until a pellicle begins to be formed. Then stir the contents with a glass rod while continuing the evaporation until a granular salt remains. Should this not be perfectly white or colorless, redissolve it in its own weight of boiling distilled water, add a sufficient quantity of ammonia water to render the solution slightly alkaline to test-paper, boil the liquid a few minutes, filter it, and evaporate the filtrate to dryness as before.

Reaction.

$2Fe+2Br_2=2FeBr_2$; then $3FeBr_2+Br_2=FeBr_2.2FeBr_3$; then

$FeBr_2.2FeBr_3+8H_4NOH=8H_4NBr+FeO.Fe_2O_3+4H_2O$.

Description.—Colorless, transparent crystals, or a white crystalline granular salt, odorless, of pungent saline taste. Soluble in 1.50 parts of water, and in 30 parts of alcohol at 15° C.; in 0.7 parts of boiling water, and in 15 parts of boiling alcohol.

AMMONIUM CHLORIDE—PURIFIED.

$H_4NCl=53.4$.

Crude ammonium chloride	20 parts
Water	30 parts
Chlorine water	1 part
Ammonia water (10%)	1 part

Dissolve the ammonium chloride in the water heated to boiling, then add the chlorine water, stir well and continue the heating for twenty minutes. Add the ammonia water. Continue the boiling for a few minutes. Filter. Evaporate to granulation. Dry the product thoroughly.

Pure Ammonium Choride

may be readily made from pure diluted hydrochloric acid and pure ammonia water:

Diluted hydrochloric acid (10% of HCl)	2 parts
Ammonia water (10% of H_3N)	1 part

Add the ammonia water gradually to the acid, stirring well. Evaporate to granulation or crystallization, and dry the product.

Reaction. $H_3N+HCl=H_4NCl$.

Description.—Pure ammonium chloride is a colorless crystalline, or a white, granular salt; odorless, taste saline, cooling. Soluble in 3 parts of water of 15°, and in its own weight of boiling water. Nearly insoluble in alcohol. Reaction neutral. Volatilizes without residue when strongly heated.

AMMONIUM OXALATE.

$(H_4N)_2C_2O_4.H_2O=142$.

Oxalic acid	100 Gm
Boiling distilled water	800 ml
Ammonium carbonate, sufficient.	

Dissolve the acid in the water, neutralize with ammonium carbonate, raising the temperature, at the end of the neutralization, to the boiling point; filter while hot, and set aside to cool and crystallize.

Reaction. $2(H_4NHCO_3.H_4NH_2NCO_2)+3H_2C_2O_4$

$=3(H_4N)_2C_2O_4+2H_2O+4CO_2$.

Notes. About 83 Gm of ammonium carbonate will be required to saturate 100 Gm of oxalic acid. Instead of 83 Gm of ammonium carbonate, the corresponding quantity (270 ml) of ammonia water (10 per cent) may be used. The solution should be rendered exactly neutral to test-paper, filtered while hot, and set aside to cool slowly. When the solution is dilute and the cooling and crystallization slow, very handsome needle-shaped crystals may be obtained. By evaporating the mother liquor and again crystallizing, additional crops of the salt are recovered.

Description.—This salt crystallizes in rhombic prisms, easily soluble in water.

AMMONIUM SULPHATE.

$(H_4N)_2SO_4=132$.

Ammonia water	2 volumes
Diluted sulphuric acid	5 volumes

Add the acid gradually to the ammonia water, stirring the liquid. Should the solution not be alkaline in its reaction on test-paper, add enough additional ammonia water to render it alkaline. Filter. Evaporate to crystallization. During the evaporation test the liquid from time to time with litmus paper, and add more ammonia if necessary to prevent the solution from acquiring an acid reaction, or to render it alkaline again should it become acid by the loss of ammonia.

Reaction. $2H_4NOH+H_2SO_4=(H_4N)_2SO_4+2H_2O$.

Description.—Colorless and odorless crystals, soluble in 1.33 parts of water at 15°.

SOLUTION OF ANTIMONY CHLORIDE.

A solution of $SbCl_3$ in water containing hydrochloric acid.

Purified antimonous sulphide, in extremely fine powder	100 Gm
Hydrochloric acid (35% of HCl)	530 Gm

Put the acid in a flask of one cubic-decimeter's capacity. Heat it to about 50°. Add the antimonous sulphide, *in very fine powder,* gradually to avoid too copious evolution of hydrogen sulphide. Shake well. Apply heat, first gently, then gradually raising the temperature to the boiling point of the liquid. Boil for half an hour. Let the liquid cool somewhat. Filter it through muslin, returning the portion which first passes until the filtrate runs clear. Evaporate the filtrate to 250 ml, and put it in a glass-stoppered bottle.

Reaction. $Sb_2S_3+6HCl=2SbCl_3+3H_2S$.

Notes. A very large excess of hydrochloric acid is used in order to hold the antimony chloride in solution. Nevertheless, the action of the acid upon the antimony sulphide is slow. A high temperature is necessary. The operation should be carried out under a hood with a good draught to carry off the vapors of hydrogen sulphide.

Some hydrochloric acid is lost by volatilization during the process of heating it with the antimony sulphide. A hydrochloric acid of less than 35 per cent (1.17 sp. w.) strength does not dissolve the antimony sulphide completely.

The antimonous sulphide should be *pure,* and in very fine ("impalpable") powder. It may be tested before being used, to see that it is soluble in the strong hydrochloric acid without any residue.

The purified antimonous sulphide, although nearly or quite free from arsenic, may contain some lead.

The proportions of antimony sulphide and hydrochloric acid are those prescribed in the British Pharmacopœia of 1885.

Description.—A yellowish-red liquid of the odor of hydrochloric acid. It gives a white precipitate when mixed with water. Sp. w. about 1.47; sp. vol. 0.680. It can be diluted with 1.25 times its own weight of water without permanent precipitation.

ANTIMONY OXIDE.

Sb_2O_3=285 (or Sb_4O_6=570).

Solution of antimony chloride	250 ml
Ammonia water (10%)	115 ml
Water.	

Add just enough water to the solution of antimony chloride to produce a slight but permanent turbidity. Boil the liquid for about five minutes. Filter. Pour the filtrate slowly into 2500 parts of water contained in a glass or stoneware vessel, stirring the mixture diligently during the addition of the antimony solution to the water. Let the precipitate subside.

Siphon off the supernatant liquid and add to the precipitate 2500 parts of hot water, stir well, let stand until the precipitate has settled, then draw off the wash water. Repeat this washing once more, in the same manner, with another 2500 parts of hot water, and siphon off the liquid. Then add the ammonia water to the precipitate, let the mixture stand for an hour, stirring frequently. Collect the precipitate on a filter and wash it with boiling water until a test-portion of the washings, after acidulation with nitric acid, no longer produces a cloudiness with test-solution of silver nitrate. Then drain the oxide and dry it at a temperature not exceeding 100°.

Reaction.

$4SbCl_3+5H_2O=2SbOCl.Sb_2O_3+10HCl$; and, finally,

$2SbOCl.Sb_2O_3+2H_4NOH=2Sb_2O_3+2H_4NCl+H_2O$.

Notes. Any lead chloride present in the solution of antimony chloride is washed out from the precipitated "oxychloride of antimony" by means of hot water.

Sodium carbonate may be used instead of ammonia water (using 95 parts of the carbonate in place of 115 parts of ammonia water).

Description.—A heavy, light-grayish, or nearly white, odorless and tasteless powder, insoluble in water and in alcohol. It turns yellow when heated, but becomes white again on cooling.

ANTIMONYL-POTASSIUM TARTRATE.

[Tartar Emetic.]

$2KSbOC_4H_4O_6.H_2O=660.42$.

Antimonous oxide	4 parts
Potassium bitartrate	5 parts
Water	50 parts

Mix the oxide and the cream of tartar with enough water to form a paste, and set this aside for twenty-four hours. Then add the remainder of the water and boil the mixture in a porcelain capsule for fifteen minutes, stirring frequently, and replacing the water lost by evaporation. Filter while hot, evaporate the filtrate to 30 parts, and set it aside to crystallize. Wash the crystals with a little cold water, and then dry them between filter paper.

Reaction. $2KHC_4H_4O_6+Sb_2O_3=2K(SbO)C_4H_4O_6+H_2O$.

Notes. A slight excess of antimonous oxide is used, but remains undissolved and is filtered away. To obtain the salt in minute crystals the solution obtained by the above formula may be evaporated down to 27 parts and then shaken in a bottle until cold, when the greater part of the salt will separate. The mother liquor will yield more crystals, but after two or three crystallizations it becomes quite colored, as is the case in making several other tartrates.

Description.—Small, transparent crystals, or white powder, odorless, having a sweet, afterwards disagreeable, metallic taste. Soluble in 17 parts of water at 15°, and in 3 parts of boiling water. Insoluble in alcohol.

The colorless crystals become white or opaque on exposure to the air.

ANTIMONIC SULPHIDE.

(*Stibium Sulphuratum Aurantiacum.—"Golden Sulphuret of Antimony."*)

Chiefly $Sb_2S_5=398$.

Purified black antimony sulphide......	18 parts
Sublimed sulphur	5 parts
Sodium hydroxide	13 parts
Water	50 parts

Sulphuric acid, distilled water, each sufficient.

Triturate the antimonous sulphide and the sulphur together until mixed; add the mixture to the sodium hydroxide previously dissolved in the water; boil the mixture with constant stirring about fifteen minutes, or until no more of the powder dissolves. Filter, evaporate the filtrate, and let it cool, that crystals may form.

Rinse the crystals hastily with a little weak soda solution, and dry them by pressing them between bibulous paper. [These crystals are the so-called "Schlippe's Salt"—$Na_3SbS_4+9H_2O$.]

Dissolve 100 parts of these crystals in 400 parts of distilled water. Filter, if necessary. Dilute the filtrate with six hundred parts of distilled water, and pour the dilute solution slowly and with constant stirring into a mixture of thirty-six parts (by weight) of sulphuric acid and six hundred parts of distilled water.

Wash the precipitate as rapidly as possible, by affusion and decantation, with distilled water, let drain, express the remainder of the water, and dry the precipitate with the aid of gentle heat. Keep it in a tightly corked bottle, protected from light.

Reaction.

$$4Sb_2S_3+4S_2+18NaOH=5Na_3SbS_4+3NaSbO_3+9H_2O;$$

then, $2Na_3SbS_4+3H_2SO_4+10H_2O$

$$=Sb_2S_5+3Na_2SO_4.10H_2O+3H_2S.$$

Notes. The direction to wash the crystals of Schlippe's salt is given for the purpose of removing sulpharsenate, which is more readily soluble, and a little sodium hydroxide is added to the wash-water in order to prevent oxidation.

The sulphuric acid must be in excess; otherwise the product will be dark colored.

The washing must be done with distilled water, and should not be long continued. Affusion and decantation is therefore to be perferred.

Several formulas prescribe an excessive proportion of sulphur—equal weights of sulphur and antimony sulphide.

Description.—An orange-red, odorless, insoluble powder.

BARIUM CHLORIDE.

$BaCl_2.2H_2O=243.56$.

Powdered witherite	10 parts
Hydrochloric acid (31.9% of HCl)	11 parts
Barium sulphide.	
Water.	

Mix the witherite thoroughly with its own weight of water in a porcelain dish. Then add, gradually, the hydrochloric acid previously mixed with 10 parts of water, stirring well, waiting after each addition of the dilute acid until the effervescence has subsided before adding more. When the interaction becomes tardy, heat the dish over the water-bath to facilitate the saturation of the acid. When all of the acid has been added, evaporate the unfiltered liquid to dryness, and heat the dry residue at about 100° for half an hour. Then add 50 parts of boiling water; stir well, continuing the heating and stirring about five minutes. Filter. Add to the filtrate a solution of barium sulphide until no further precipitation is produced by it. Filter again. Acidify the filtrate with hydrochloric acid so that it just shows a distinct acid reaction on litmus paper. Then evaporate the solution to the density of about 1.3 and set it aside in a cool place to crystallize.

Recrystallize the salt two or three times, as may be necessary.

Reaction. $BaCO_3+2HCl=BaCl_2+H_2O+CO_2$.

Notes. The mineral "witherite" is an impure native barium carbonate, containing varying amounts of calcium, strontium and iron compounds together with other mineral substances. A very large excess of witherite must necessarily be used, because much of it may consist of substances other than barium carbonate, and it is further requisite, that, after the saturation of the acid, a considerable amount of barium carbonate shall still remain in order that, during the evaporation of the solution to dryness, the iron and calcium may be precipitated. The remaining iron and any other heavy metals present are removed by precipitation with barium sulphide, which must, however, be employed with great caution to avoid using an excess. The final solution still contains chlorides of the alkali metals and of calcium and strontium, which must be removed by recrystallization of the barium chloride which is less freely soluble than the others, so that the latter remain in the mother liquors. The concentrated solution of barium chloride can also be mixed with alcohol, which precipitates the salt while the calcium and strontium chlorides remain in solution.

A pure barium chloride may, of course, be at once obtained by saturating pure hydrochloric acid with pure barium carbonate.

Description.—Colorless crystals, soluble in 3 parts of water at 10°, in 2 parts at 72°, and in 1.6 parts of boiling water. The solution is neutral.

BARIUM NITRATE.

$Ba\ (NO_3)_2 = 261$.

Powdered witherite..................	16 parts
Nitric acid (68% of HNO_3)...........	15 parts
Water.	

Mix the witherite thoroughly in a porcelain dish with its own weight of water. Dilute the nitric acid with its own weight of water. Add the diluted acid, in small portions, to the witherite, stirring well and waiting after each addition until the effervescence has subsided before adding the next portion of acid. When about one-half of the acid has been added, place the dish over a water-bath, apply heat, add about 40 parts of hot water, stir well and then proceed with the addition of the remainder of the nitric acid, in small portions, as before, stirring well. Evaporate the contents of the dish to dryness. Add to the dry residue 60 parts of boiling water, stir well, and continue heating the mixture at 90° or over for half an hour. Filter. Acidify with nitric acid. Evaporate the filtrate to crystallization. Recrystallize two or three times, as may be necessary.

Reaction. $BaCO_3+2HNO_3=Ba(NO_3)_2+H_2O+CO_2$.

Notes. The impure barium carbonate is used in large excess (see notes under Barium Chloride). Iron is precipitated by the excess of barium carbonate. The nitrates of strontium and calcium remain in the mother-liquors when the product is recrystallized.

The crystallization proceeds most satisfactorily when the acidified solution saturated at about 80° to 90° is slowly cooled.

Pure barium carbonate and nitric acid readily produce a pure barium nitrate.

Description.—Colorless or white crystals. Soluable in 7 parts of water at 10°; in 9.2 parts at 20°; in 5 parts at 60°; and in a little less than 3 parts of boiling water.

BISMUTHYL NITRATE.

Chiefly $OBiNO_3.H_2O=303$.

Bismuth, in small pieces	500	Gm
Nitric acid	1,500	ml
Sodium carbonate	2,500	Gm
Ammonia water	1,250	ml
Distilled water, sufficient.		

Mix 800 ml of the nitric acid with 1,000 ml of the distilled water in a capacious glass vessel, add the bismuth, and set aside for twenty-four hours to dissolve. Dilute the solution with 2,500 ml of distilled water, so that the turbidity produced by the addition of the water no longer disappears on stirring; stir well, and again set it aside for twenty-four hours. Then filter.

Dissolve the sodium carbonate in 5,000 ml of distilled water, with the aid of heat, filter the solution, and let it rest until cold. Then add the cold solution of bismuth nitrate very slowly, and with constant, active stirring, to the sodium carbonate solution.

Transfer the whole to a wetted muslin strainer, let drain, wash with distilled water until the washings pass tasteless, and then let it drain again as completely as possible.

Put the moist precipitate in a suitable vessel, gradually add the remainder of the nitric acid, being careful to avoid foaming over, and when all has dissolved, also add gradually 1,000 ml of distilled water, and set the solution aside for twenty-four hours. Then filter.

Dilute the filtrate by adding gradually 6,000 ml of distilled water, stirring well. Then add to this, very slowly, the ammonia water, through a small siphon, stirring constantly. Transfer the whole at once to a strainer, and after draining rapidly, pour over the precipitate 8,000 ml of distilled water, let it drain again, and press out as much of the liquid as possible.

Then dry the product on bibulous paper with a gentle heat, and rub it into powder.

Reaction. First the bismuth is dissolved in the nitric acid to form normal bismuth nitrate, thus:

$$2Bi+8HNO_3=2Bi(NO_3)_3+N_2O_2+4H_2O.$$

Then bismuth subcarbonate is made:

$$\begin{aligned}&2Bi(NO_3)_3+3Na_2CO_3.H_2O\\&\qquad=(BiO)_2CO_3.H_2O+6NaNO_3+2CO_2.\end{aligned}$$

The bismuth subcarbonate is next re-dissolved in nitric acid to form normal bismuthous nitrate again:

$$(BiO)_2CO_3.H_2O.+6HNO_3=2Bi(NO_3)_3+4H_2O+CO_2.$$

Finally the normal bismuthous nitrate is converted into subnitrate as follows:

$$Bi(NO_3)_3+2H_3N+2H_2O=OBiNO_3.H_2O+2H_4NNO_3.$$

Notes. The metal dissolves rapidly at first, but toward the last it is best to apply heat if economy of time is desired. Perforated baskets of stoneware are used for suspending the bismuth in the acid when bismuth preparations are prepared on a moderately large scale, the acid being placed in a stone pot and the basket containing the bismuth suspended just below the surface of the acid. Unless the vessel in which the solution is effected is sufficiently large to obviate all risk and inconvenience from the violence of the reaction, the metal should be added in portions, each portion to be entirely dissolved before more is added. The capacity of the vessel ought to be about three times the combined volume of the acid and water used to dissolve the metal.

The strong solution can not be filtered through paper, for the acid liquid is so corrosive as to destroy the paper, not only defeating the filtration but also discoloring the solution. It is best to filter through coarsely powdered glass or glass wool, or washed asbestos; sometimes the solution is filtered through cotton previously immersed in dilute nitric acid. Decantation is the easiest method, and generally sufficient, especially when large quantities are operated upon. After dilution, however, filtration through paper is entirely practicable.

The object of first preparing subcarbonate of bismuth is to get rid of the arsenic which the metal contained. This arsenic is held in the acid solution as bismuth arsenate, nearly all of which precipitates upon dilution of the liquid with water, and is removed by filtration after standing twenty-four hours. The remainder of the arsenic is left in the last mother liquor as ammonium arsenate, together with the ammonium nitrate after precipitation of the bismuth subnitrate with ammonia.

The character and composition of subnitrate of bismuth depend greatly upon the method of its preparation. Among the most important influences that effect the character of the product are these: The relative quantity of water used in the precipitation; the length of time the precipitate is exposed to the mother liquor; the amount and temperature of the water used for the precipitation; the quantity of water and the length of time consumed in washing; the use or omission of ammonia; and the greater or less care bestown upon the various details of the process.

The process given above probably yields the most uniform and satisfactory results. In order to insure success each step of the process should be carried out in strict accordance with the precise directions given.

In order to obtain a good product, the nitric acid used must be pure, distilled water must be used, and the acid solutions must not come in contact with wood or other organic substances, or with metals.

Whenever the precipitated bismuth subnitrate is permitted to remain long in contact with the mother liquor in which it has been thrown down, it becomes more dense, and at the same time takes up more nitric acid. This must be avoided. Long contact with much water also renders the preparation heavier, but at the same time more basic. Cold liquids will yield a voluminous precipitate; hot water makes the precipitate more dense. If the water used for precipitating is 50°C. the precipitated subnitrate will have a composition corresponding to about 77 per cent of Bi_2O_3; if the water is boiling hot (100°C.) the subnitrate will have a composition corresponding to about 80 to 81 per cent of Bi_2O_3. According to Loewe, the precipitate is not affected by a solution of ammonium nitrate, and hence it can be washed with such a solution without danger.

The precipitate must be dried at a low temperature (not over 30° C.), but must be thoroughly dry before it is put away. After removing all water that can be gotten rid of by pressing it, the precipitate should be dried in layers. If not thoroughly dried it decomposes by the influence of light and acquires a nitrous odor. If dried properly in layers, these can be easily broken and powdered by rubbing the pieces through a fine wire sieve by means of a soft brush. If dried at too high a temperature the pieces are hard, not so readily powdered, and apt to yield a coarse, harsh powder.

Good subnitrate of bismuth is soft and bulky.

Description.—A fine, white powder, odorless, almost tasteless; insoluble in water, alcohol, and glycerin.

Bismuth subnitrate is always a comparatively heavy substance, but it must be as bulky as it can be made. The product varies extremely in this particular. A relatively very heavy and coarse preparation is not fit for medicinal uses.

BISMUTH SUBGALLATE.

Crystallized normal bismuth nitrate....	100 Gm
Glacial acetic acid....................	200 Gm
Gallic acid	33 Gm

Dissolve the bismuth nitrate in the acetic acid and dilute the solution with 500 Gm of distilled water. Filter. Add gradually to this liquid a solution of the gallic acid in 1,500 Gm of cold distilled water. Wash the precipitate with tepid distilled water until the washings are free from nitric acid. Dry the product at from 70° to 80° C. on blotting paper spread upon porous tiles.

Description.—A sulphur-yellow, odorless and tasteless powder; insoluble in water or in alcohol. Probably practically identical with the preparation called "dermatol."

CALCIUM BENZOATE.

$Ca(C_7H_5O_2)_2.4H_2O=354.$

Lime	1 part
Benzoic acid	4 parts
Water	150 parts

Slake the lime in a porcelain dish with a part of the water. When the reaction has been completed add the remainder of the water, mix thoroughly, and then add the benzoic acid. Boil the mixture a few minutes until the benzoic acid is dissolved. Filter the solution while hot. Evaporate the filtrate to one-half its volume and set it aside to cool. Collect the crystals and drain and dry them. Recover the remainder of the salt from the mother-liquor by evaporation in the usual way.

Notes. Very large, long crystals can be obtained by spontaneous evaporation of a saturated solution. The crystals may be dried with the aid of gentle heat. When the mother-liquor ceases to yield enough to warrant further evaporation, the benzoic acid may be recovered from it by precipitation with a sufficient amount of hydrochloric acid.

Description.—Long, white needles, soluble in about 25 parts of water at 15°, and in a smaller proportion of boiling water.

CALCIUM CARBONATE; PRECIPITATED.

$CaCO_3=100.$

Calcium chloride, fused................	10 parts
Sodium carbonate	26 parts
Distilled water, sufficient.	

Dissolve the calcium chloride in 100 parts of the water, and the sodium carbonate in another equal quantity. Filter the solutions. Heat both solutions to about 80° C. Pour the solution of calcium chloride into the solution of sodium carbonate, stirring constantly. Wash the precipitate by decantation; then collect and dry it.

Reaction. $CaCl_2+Na_2CO_3=CaCO_3+2NaCl.$

Notes. Hot solutions are used to insure a dense precipitate, so that the process of washing may be easier; when cold solutions are used the precipitated calcium carbonate is a very bulky magma, difficult to wash free from sodium chloride.

The precipitate should be washed until the washings cease to be rendered turbid by test solution of silver nitrate, and then dried at 100° C.

Description.—A very fine, perfectly white powder, odorless, tasteless, and insoluble in water and in alcohol. Under the microscope the precipitated calcium carbonate is seen to be micro-crystalline.

CALCIUM CHLORIDE.

$CaCl_2=110.8$.

White marble, in powder	250 Gm
Hydrochloric acid (31.9% of HCl)	500 ml
Water	500 ml
Chlorine water.	
Lime.	

Mix the acid and water. Add the marble in small portions, stirring well, and let the effervescence subside after each addition before adding more. When all the marble has been added and the reaction has nearly ceased, heat the mixture to boiling until no more marble dissolves, replacing the water lost by evaporation. Add to the hot liquid 15 ml of strong chlorine water, stir well, and continue boiling for half an hour. Add milk of lime a sufficient quantity to render the liquid distinctly alkaline. Filter while hot. Evaporate to dryness in a tared dish and fuse the residue at a low red heat.

Keep the product in tightly closed bottles.

Reaction. $CaCO_3+2HCl=CaCl_2+H_2O+CO_2$.

Notes. Marble is liable to contain some iron. Used in excess it reprecipitates dissolved iron and other metals. The chlorine water has for its object the oxidation of the iron to the ferric condition to facilitate precipitation, which is further insured by the addition of milk of lime.

Should the product still contain iron after fusion, it must be redissolved in twice its weight of boiling water, again treated with chlorine water, and also with either milk of lime or pure calcium carbonate, the hot solution filtered, and again evaporated to dryness and the residue fused. In evaporating to dryness add hydrochloric acid occasionally to prevent the product from becoming alkaline.

Description.—Hard white pieces or masses; odorless; taste saline, acrid. Highly deliquescent. Soluble in 1.5 parts of water at 15° and in 8 parts of alcohol; in 1.5 parts of boiling alcohol and freely in boiling water.

CALCIUM PHOSPHATE.

[Precipitated Tri-Calcium Phosphate.]

$Ca_3(PO_4)_2=310.$

Bone-ash, in fine powder	300 Gm
Hydrochloric acid	450 ml
Ammonia water, sufficient.	

Digest the bone-ash with the acid, diluted with 1,500 ml of water, until dissolved. Boil the solution a few minutes; filter; then add 1,500 ml of boiling water. Now add ammonia water until the mixture acquires an alkaline reaction. Collect the precipitate on a muslin strainer, wash it with hot water until the washings cease to produce a precipitate with test solution of silver nitrate acidulated with nitric acid. Dry the washed precipitate at a temperature not exceeding 100° C.

Reaction. When the crude tri-calcium phosphate is dissolved in hydrochloric acid, the reaction occurring is as follows:

$$Ca_3(PO_4)_2+4HCl=CaH_4(PO_4)_2+2CaCl_2.$$

When ammonia is added to the solution both the acid calcium phosphate and the calcium chloride are decomposed,

$$CaH_4(PO_4)_2+2CaCl_2+4H_4NOH+$$
$$4H_4NCl+4H_2O+Ca_3(PO_4)_2.$$

CHROME ALUM.

$KCr(SO_4)_2.12H_2O=499.$

Potassium dichromate	4 parts
Sulphuric acid (92.5% of H_2SO_4)	5 parts
Water	9 parts
Starch	1 part

Put the water in a large enough porcelain dish to hold at least twice the total quantities operated upon. Set the water into rapid rotary motion by stirring with a glass rod. Add the sulphuric acid in a thin stream. Dissolve the potassium dichromate in the mixture. Then add the starch a small quantity at a time, stirring cautiously. When the reaction is nearly ended, heat the liquid, *if necessary,* until it acquires a bluish-green color. Filter the solution and set it in a cold place to crystallize.

Reaction.

$$2K_2Cr_2O_7+8H_2SO_4+3C+40H_2O=4[KCr(SO_4)_2.12H_2O]+3CO_2.$$

Notes. The temperature of the reaction is very high, and the chemical interaction liable to be violent unless the starch be added very slowly. It is necessary that the mixture should remain hot throughout the whole reaction, but no application of heat from without is necessary, nor should it be resorted to until after the violence of the action has subsided. The liquid must at the end be bluish-green, and not olive-green. Should the bluish-green color be attained without the application of heat, no heating is necessary. Should, on the other hand, the liquid be olive-green, heat it cautiously until it acquires a bluish-green color.

In case the crystallization should be much retarded, evaporate the solution somewhat, and then set it in a cold place. Should it still fail to crystallize drop into it a few crystals of chrome alum to start the crystallization.

As the salt is efflorescent it must be kept in tightly closed bottles in a cool place.

Description.—Dark purple, transparent crystals, soluble in 7 parts of water at 15°. The cold solution is reddish-blue, but above 70° it becomes bluish-green.

COPPER SULPHATE.

$CuSO_4.5H_2O=249.5$.

Copper, in filings	5 parts
Sulphuric acid	8 parts
Nitric acid	4 parts
Water	30 parts

Mix the ingredients in a flask and heat at first gently, and afterwards raise the temperature gradually to the boiling point. Boil until the evolution of gas ceases, evaporate the solution to dryness, dissolve the residue in four times its weight of water, filter and evaporate to crystallization. Dry the crystals without the aid of heat.

Reaction. $3Cu_2+6H_2SO_4+4HNO_3=6CuSO_4+2N_2O_2+8H_2O$.

Precipitated copper sulphate may be made by dissolving the salt in its own weight of boiling water, and filtering the solution into an equal volume of alcohol.

Turbidated copper sulphate may be made by dissolving 20 parts of coarsely powdered copper sulphate in 20 parts of boiling water, acidifying with 1 part of diluted sulphuric acid, filtering the solution while hot, and stirring the filtrate briskly until cold. The granular salt thus obtained is collected on a filter, drained, dried at the ordinary temperature, and bottled. The mother liquor is evaporated to one-half its weight and an additional amount of product obtained from it in the same manner. All of the remaining salt may be recovered by evaporation nearly to dryness, and finishing the drying by exposure to the air.

Description.—Copper sulphate consists of large, transparent, clear, deep-blue crystals; or a blue, granular, crystalline, coarse powder if granulated, precipitated by alcohol, or turbidated. It is odorless, and has a nauseous, strongly metallic taste. Slowly loses its water of crystallization in dry air.

Soluble, at 15° C., in about 2.6 parts of water, and in 0.5 part of boiling water; almost insoluble in alcohol.

When carefully and continuously heated to 30° C., the salt loses 2 of its 5 molecules of water (14.43 per cent), and is converted into a pale-blue, amorphous powder. Two more molecules of water are lost at 100° C., while the fifth is retained until 200° C. is reached, when a white, anhydrous powder remains (63.9 per cent of the original weight). At a still higher temperature sulphur dioxide and oxygen are given off, and a residue of black cupric oxide is left.

The aqueous solution (1 in 20) has a blue color and shows an acid reaction on litmus paper.

AMMONIATED COPPER.

Copper sulphate	3 parts
Ammonium carbonate	4 parts

Crush the salts; triturate them together in a porcelain mortar.

Notes. The reaction between the salts causes the mixture to become wetted by the water of crystallization liberated from the decomposed copper sulphate. The mixture acquires a deep-blue color. An "ammoniacal" copper sulphate is formed, the composition of which is uncertain. Effervescence takes place from the separation of CO_2.

When effervescence has ceased and the mixture has been reduced to a dry powder this product is to be kept in a tightly stoppered bottle.

IRON (FERRIC) CITRATE.

Evaporate any convenient quantity of the solution of ferric citrate over a water-bath at a temperature not exceeding 60° C. to the consistence of syrup, and spread it on glass plates to dry in scales.

Keep it in well stoppered bottles in a cool, dark place.

Description.—Thin, transparent, garnet-red scales, odorless, of slightly ferruginous and only slightly acidulous taste. Freely soluble in water; quickly in hot water. Insoluble in alcohol. It gradually loses its ready solubility in water, and more rapidly when exposed to light. The percentage of iron is from 19 to 20%, but it varies; and the percentage of moisture retained by the scales is also variable. The preparation nearly corresponds to $FeC_6H_5O_7.3H_2O$, but may contain less iron and more water than that formula indicates. It is, therefore, not a definite chemical compound.

Ammonio-Ferric Citrate.

Solution of ferric citrate................	3 parts
Ammonia water	1 part

Mix and evaporate the mixture over a water-bath at not over 60° C. to the consistence of thick syrup; spread this on glass plates and let it dry in scales.

Keep the product in a well stoppered bottle, in a cool, dark place.

Description.—Thin, transparent, garnet-red scales; odorless; taste slightly saline and faintly ferruginous. Completely and quickly soluble in water. Insoluble in alcohol.

IRON (FERRIC) CITRATE SOLUTION.

LIQUOR FERRI CITRATIS; U. S. P. 1890.

Solution of normal ferric sulphate......	84 parts
Citric acid	30 parts
Ammonia water (10% of H_3N)........	90 parts
Distilled water.	

Mix the ammonia water with 300 parts of cold water, and the iron solution with 1,000 parts of cold water. Add the cold diluted iron solution gradually to the ammonia solution, stirring constantly. Let the precipitated ferric hydroxide subside, and decant the supernatant liquid. Pour the remaining liquid, containing the magma, upon a wetted muslin strainer; let it drain;

return the precipitate to the precipitation vessel and mix it well with 1,500 parts of cold water. Let settle, decant, and again drain the magma on the muslin strainer. Repeat this washing several times, until the washings give but a slight cloudiness when mixed with test-solution of barium chloride. Finally let the ferric hydroxide be well drained. Then place it in a strong press cloth and forcibly express from it, by means of a screw press, as much as possible of the water. [The magma obtained from 84 parts of solution of ferric sulphate may be pressed until the press cake weighs less than 65 parts.]

Put the citric acid in a porcelain dish and add to it about one-half its weight (15 parts) of distilled water. Place the dish over a water-bath and keep its contents at a temperature not exceeding 40°. Add the press cake of ferric hydroxide, in small portions at a time, stirring well, and allowing each portion to dissolve before adding another. When about three-fourths of the ferric hydroxide has been added, raise the temperature of the contents of the dish to 50°, and when all of the ferric hydroxide has been added let the temperature be increased to 60°, and continue heating and stirring for about fifteen minutes, taking care not to allow the temperature to rise above 60°. Filter the solution and evaporate it at a temperature not exceeding 60° until it weighs 100 parts.

Keep the product in well closed bottles in a cool place and protected from light.

An equivalent amount of solution of ferric sulphate of the present Pharmacopœia is substituted for that of 1890.

Reactions.

$Fe_2(SO_4)_3+6H_4NOH=2Fe(OH)_3+3(H_4N)_2SO_4$; then

$Fe(OH)_3+H_3C_6H_5O_7=FeC_6H_5O_7+3H_2O$.

Notes. The pharmacopœial directions do not require that the water should be squeezed out of the magma of ferric hydroxide, nor that the hydroxide shall be added gradually to the citric acid mixed with a little water. The Pharmacopœia of 1890 instead directs that the drained magma be at once mixed with the citric acid and the mixture heated at 60°

until the precipitate (the hydroxide) is dissolved. It is better, however, to proceed as here described, adding gradually the ferric hydroxide, deprived of as much water as can be pressed out of it, and raising the temperature to 60° only at the end. By proceeding in this manner the saturation of the citric acid will be affected without exposing the ferric hydroxide or the solution of ferric citrate to that high temperature any longer than is unavoidable, and the concentration of the solution to the required standard requires but little evaporation.

Ferric hydroxide is easily decomposed and rendered insoluble, or difficultly soluble, by heat. But a higher heat than the temperature of the room is necessary to effect the solution of the ferric hydroxide in the citric acid solution, and especially so when saturation is approached.

If 1 ml of ammonia water of ten per cent (H_3N) strength be added to the citric acid and water before the ferric hydroxide is added, the solution of the latter is greatly hastened and this small quantity of ammonia should not be objected to, amounting to only 1 Gm of H_3N in each liter of finished product.

A small amount of the ferric hydroxide remains undissolved, and is filtered away before the solution is evaporated to 100 Gm. This undissolved hydroxide is usually basic owing to the exposure of the normal hydroxide to heat.

Examinations made of many samples of solution of ferric citrate and of the ferric citrate in scales show that the percentage of iron in these preparations is quite variable. This is due to the varying degree of care observed in their manufacture, and especially to the conversion of a greater or less portion of the ferric hydroxide into insoluble meta-hydroxide. It is intended that the citric acid shall be saturated with ferric hydroxide, but the fact that a portion of the hydroxide remains undissolved at the end does not indicate saturation if the undissolved portion consists of basic ferric hydroxide for that is insoluble. Hence it is necessary in order to obtain as nearly uniform results as practicable, to adopt all the precautions described in the working directions given in the foregoing formula.

The Pharmacopœia of 1890 states that the solution is of a strength corresponding to about 7.5 per cent of metallic iron. This is a minimum of iron. When well made the solution must contain about 35.5 per cent of normal ferric citrate, that being the proportion of ferric citrate formed by the quantity of citric acid employed. Assuming that the citric acid is all neutralized so as to form normal ferric citrate, the finished preparation contains about 8 per cent of iron. One hundred grams of the solution should, upon evaporation, yield about 42.5 per cent of scaled ferric citrate; but the yield depends in part upon how far the solution is evaporated before it is put on the glass plates to be dried, and the temperature at which the scaling is effected, or, in other words, the amount of moisture retained by the scaled salt. If the scaled salt corresponds to $FeC_6H_5O_7.3H_2O$, the yield from 100 Gm of solution can not be over 42.46 Gm.

Description.—The solution of ferric citrate is dark brown-red, odorless, and has a slightly ferruginous and only faintly acid taste. Its reaction on test-paper is acid. The sp. w. is not less than 1.250 at 15°.

IRON (FERRIC) HYDROXIDE.

$Fe(OH)_3=107$.

Solution of normal ferric sulphate (40 Gm, or)	28 ml
Ammonia water (40 Gm, or)	42 ml
Water, sufficient.	

Dilute the ammonia water with 100 ml of cold water in a precipitation vessel of about one liter's capacity. Dilute the solution of ferric sulphate with 500 ml of cold water, and pour this into the dilute ammonia slowly and during constant stirring.

Wash the precipitated ferric hydroxide with cold water, first by affusion and decantation, and afterwards on a wetted muslin strainer, until the washings are tasteless. Then let the magma drain, return it to the precipitation vessel, and mix it well with as much cold water as the vessel will hold, or about one liter. Let settle, decant the supernatant liquid, and again transfer the magma to the wetted strainer and allow it to drain. Continue the washing in this manner until the wash water is no longer affected by test-solution of barium chloride.

Reaction. $Fe_2(SO_4)_3+6H_4NOH=2Fe(OH)_3+3(H_4N)_2SO_4$.

Notes. *Dilute* and *cold* solutions are necessary. The diluted solution of ferric sulphate should be poured slowly, or in a small stream, into the diluted ammonia water during constant stirring, as directed. When the whole of the iron solution has been added, the mixture should still have a decidedly ammoniacal odor; as the ammonia gas is diffused through the stratum of air above the surface of the liquid, that air must be fanned or blown away before the odor of the liquid itself can be ascertained.

There are several ferric hydroxides, differing from each other in the proportions of HO they contain, in color, and in their solubility in acids. The product intended by the Pharmacopœia is $Fe(OH)_3$; another ferric hydroxide is $Fe_2O(OH)_4$; and dried ferric hydroxide is $OFe(OH)$. The more basic it is, and the less water it contains, the less readily soluble is it. As obtained by the official process, all the necessary precautions being observed, the product is dark reddish-brown and readily soluble in citric acid, and in glacial acetic acid. When yellowish-brown, brown, grayish-brown, or clay-colored, it is basic, insoluble and unfit for the uses for which it is intended in pharmacy.

In summer it is best to use ice for cooling the dilute solutions previous to the precipitation. The chemical reaction generates heat, and if the solutions are already warm the temperature of the mixture is liable to rise sufficiently high to cause the formation of light-colored meta-hydroxide. But the liquids must not be ice-cold.

Should the quantity of ammonia used be insufficient, or should it be added to the ferric sulphate instead of *vice versa,* basic ferric salts will be formed. If the H_3N is added to the iron solution a dark red-brown solution containing ferric salt is obtained at first, and no ferric hydroxide is thrown down until more ammonia is added.

Ferric hydroxide may also be precipitated from ferric chloride or ferric nitrate, and soda may be used as a precipitant instead of ammonia.

If solution of ferric chloride is used the hydroxide formed is more easily washed free from the ammonium salts. If ferric nitrate is used, the product is soluble in an excess of potassium carbonate (Stahl's tincture).

Ferric hydroxide precipitated with soda instead of ammonia is lighter in color and extremely difficult to wash free from sodium salt.

Ferric hydroxide forms very soluble compounds, with sugar and with glycerin. It is also soluble in solutions of ferric salts, as in solution of ferric chloride. The precipitation of ferric hydroxide from solutions of ferric salts by means of alkalies is prevented by the presence of citric acid, tartaric acid, sugar, glycerin, and certain other organic substances.

Description.—When moist it is a dark red-brown magma, insoluble in water, but soluble in citric, tartaric, acetic, hydrochloric, sulhpuric, and nitric acids. When dried it is a reddish-brown amorphous powder, less readily soluble in acids than the moist hydroxide.

Uses. For the preparation of ferric acetate, citrate, tartrate, nitrate, etc. Also as an antidote for arsenic, with which it forms insoluble arsenate:

$$4Fe(OH)_3+As_2O_3=Fe_3(AsO_4)_2+Fe(OH)_2+5H_2O.$$

For whatever purpose it may be required it must be freshly prepared, as it cannot be preserved from decomposition by which meta-hydroxides are formed, which in no case can take the place of the freshly precipitated ferric hydroxide.

IRON (FERROUS) OXALATE.

$FeC_2O_4.H_2O=162.$

Ferrous sulphate	330 Gm
Oxalic acid	150 Gm
Ammonia water	300 ml

Dissolve the oxalic acid in the ammonia water diluted with 2,000 ml of water; and the ferrous sulphate in 3,000 ml of hot water; filter the solutions, and then mix them. Wash the precipitate by decantation and afterwards on a filter until the washings are tasteless. Dry the product between bibulous paper, with the aid of gentle heat.

Reaction. $FeSO_4+(H_4N)_2C_2O_4=FeC_2O_4+(H_4N)_2SO_4$.

Notes. Ferrous oxalate can also be made by the process official in the U. S. P. of 1870, which prescribed ferrous sulphate dissolved in water to be added to a solution of oxalic acid. When this process is followed ferrous oxalate and free sulphuric acid are formed, the precipitate separates slowly, and a considerable loss results from the fact that the oxalate is not insoluble in the sulphuric acid. By neutralizing nearly all of the oxalic acid with ammonia, using a slight excess of oxalic acid, and mixing the solution of ferrous sulphate with the acid solution of ammonium oxalate, the precipitate falls at once, and no loss of product is sustained. If a perfectly neutral solution of ammonium oxalate is used, the product will have a dull reddish-yellow color.

To obtain a bright yellow ferrous oxalate the mother liquor in which the precipitate is formed must be decidedly acid.

Description.—A light-yellow crystalline powder; odorless; nearly tasteless. Nearly insoluble in water, and insoluble in alcohol.

IRON (FERRIC) OXIDE.

$Fe_2O_3=160$.

Heat dried ferric hydroxide strongly until it ceases to lose weight.

$$2Fe(OH)_3=Fe_2O_3+3H_2O.$$

Seven parts of dry ferric hydroxide will yield rather more than five parts of oxide. It is brown-red.

Another Method.

Heat ferrous oxalate until completely decomposed.

$$4FeC_2O_4+3O_2=2Fe_2O_3+8CO_2.$$

The product obtained from ferrous oxalate is extremely finely divided and therefore soft and of a rich, dark-brown color. This is used by jewellers to polish gold.

Magnetic Oxide of Iron.

$Fe_3O_4=232$.

Solution of ferric sulphate, U. S.	168 ml
Ferrous sulphate	60 Gm
Ammonia water (10% of H_3N)	500 ml
Water.	

Dissolve the ferrous sulphate in two liters of water and filter. Add the solution of ferric sulphate.

Dilute the ammonia water with one liter of water.

Pour the iron solution gradually into the diluted ammonia water, stirring well.

Set the mixture aside to settle. Decant the supernatant liquid from the precipitate. Then add enough water to make the whole mixture measure about three liters.

Boil this mixture about fifteen minutes, or until the brown ferroso-ferric hydroxide is changed to the nearly black ferroso-ferric oxide.

Wash the precipitate, first by affusion and decantation of hot water, and afterwards on a filter, until the washings cease to give a precipitate with test-solution of barium chloride. Then dry the product with the aid of heat.

Reaction.

$$Fe_2(SO_4)_3+FeSO_4+8H_4NOH$$
$$=Fe_2O_3.FeO+4(H_4N)_2SO_4+4H_2O.$$

Description.—A fine, heavy, brownish-black, odorless and tasteless powder.

IRON (FERRIC) OLEATE.

$Fe(C_{18}H_{33}O_2)_3=899$.

Solution of normal ferric sulphate	66 ml
White castile soap, in fine powder	150 Gm

Dilute the solution of ferric sulphate with 5,000 ml of water, and dissolve the soap in 2,500 ml of hot water. Mix the solutions. Wash the precipitated oleate twice with hot water, using about ten liters each time.

Reaction.

$$Fe_2(SO_4)_3+6NaC_{18}H_{33}O_2=2Fe(C_{18}H_{33}O_2)_3+3Na_2SO_4.$$

Notes. The yield is about 120 Gm.

Ferrous oleate can also be made by double decomposition, from ferrous sulphate and soap, but the greenish ferrous oleate soon oxidizes.

Description.—Ferric oleate is a dark red plaster-like solid. Soluble in oleic acid and in fixed oils.

IRON (FERRIC) PYROPHOSPHATE; SOLUBLE.

[Sodio-Ferric Citro-Pyrophosphate.]

Ferric citrate	5 parts
Sodium pyrophosphate	5 parts

Dissolve the citrate in 10 parts of distilled water heated on a water-bath, add the pyrophosphate, and continue the heating with constant stirring until the pyrophosphate of sodium is dissolved and the solution turns green. Evaporate at a temperature not exceeding 60° C. (140° F.) until the solution has the consistency of thick syrup; spread it on glass plates, and dry it in scales.

Notes. The preparation is difficult to scale satisfactorily. In small quantities it may be best scaled in the cold. Pyrophosphate of iron should be carefully protected against light. It is best preserved in amber-colored bottles, tightly corked, and kept in a dry, cool, dark place.

Description.—Thin, transparent, green scales; odorless; taste a little acidulous, slightly saline. Freely soluble in water. Insoluble in alcohol.

Another Method.

[After the Swiss Pharmacopœia.]

Sodium pyrophosphate	75 parts
Solution of ferric chloride, U. S. P.	126 parts
Citric acid	26 parts
Ammonia water,	
Distilled water, of each sufficient.	

Dissolve the sodium pyrophosphate in 500 parts of distilled water.

Dilute the solution of ferric chloride with 800 parts of distilled water.

Pour the iron solution a little at a time into the solution of sodium pyrophosphate, stirring uninterruptedly.

Wash the precipitate thoroughly in the usual way.

Dissolve the citric acid in 50 parts of distilled water with enough ammonia water to render the solution slightly alkaline.

Add the moist, precipitated ferric pyrophosphate to the solution of ammonium citrate, stir well, and heat at not over 50° C. until solution is effected.

Filter the solution, evaporate it, and scale the product in the usual way.

IRON (FERROUS) SULPHATE.

$FeH_2SO_5.6H_2O=278.$

Iron in the form of clean, bright wire, cut small	3 parts
Sulphuric acid	5 parts
Water, sufficient.	

Put 15 parts of water in a flask or dish and add the sulphuric acid very gradually, stirring constantly. Then add the iron. When effervescence has nearly ceased, heat the contents to boiling for about ten minutes. Filter while hot. Set aside to crystallize.

Collect the crystals and dry them on filter paper.

Evaporate the mother liquor to one-half its weight, again set aside to crystallize, and collect the second crop of crystals in the manner as the first.

Reaction. $Fe+H_2SO_4+7H_2O=FeH_2SO_5.6H_2O+H_2$.

Notes. The iron is used in excess of amount required by theory in order that no ferric salt may be formed. The hydrogen, which is liberated in the chemical reaction, has a disagreeable odor from volatile carbon, sulphur, and phosphorus compounds.

To prevent contamination with ferric salt, the filtrate may be acidulated with sulphuric acid before being set aside to cool; any ferric sulphate present will remain in the mother liquor.

The crystals should be dried as rapidly as practicable to avoid oxidation, which easily takes place when the salt is moist. To facilitate the drying the crystals may be hastily washed with a little alcohol, which evaporates rapidly.

Recrystallized Ferrous Sulphate.

Green vitriol	10 parts
Water	15 parts
Diluted sulphuric acid	1 part

Make a solution with the aid of heat. Boil it until a filtered sample is of a bluish-green color. Then filter while hot, and set the solution aside to crystallize in the usual way. Drain and dry the crystals as rapidly as possible and at once bottle the product in a dry container to be tightly closed.

Notes. The crystals must have a bluish-green and not a yellowish-green color, and they must be perfectly clear. The ferric sulphate and other ferric compounds contained in the green vitriol are removed partly by the boiling and filtration, and partly in the crystallization when the ferric sulphate present remains in the mother liquor.

Precipitated Ferrous Sulphate.

Iron wire	2 parts
Sulphuric acid	3 parts
Distilled water	12 parts
Alcohol	6 parts

Add the sulphuric acid gradually to the water, stirring well. Add the iron to the warm mixture. When effervescence has nearly ceased heat the mixture at the boiling point for about ten minutes. Filter while hot. Add a small quantity of sulphuric acid to the filtrate. Then pour the solution into the alcohol, stirring constantly.

Collect the precipitated crystalline ferrous sulphate on a filter, transfer it to bibulous paper, and dry it quickly at the ordinary room temperature, stirring the crystals often and changing the paper once or twice to facilitate rapid drying.

When thoroughly dry, put the product in dry bottles to be well corked or stoppered.

Notes. Iron is used in excess and the acid is saturated with it. Free sulphuric acid is added to the solution because the salt crystallizes best from an acid solution.

Ferrous sulphate is insoluble in alcohol, but ferric sulphate is soluble. Hence any ferric sulphate present will remain in the alcoholic liquid.

The crystals wet with alcohol instead of water dry rapidly and are moreover surrounded by an atmosphere of alcohol vapor instead of air, so that the process of drying is accomplished with far less danger of oxidation.

IRON ALUM.

[Sulphate of Iron and Ammonium. Iron Alum.]

$FeH_4N(SO_4)_2.12H_2O=482.$

Solution of normal ferric sulphate......	221 ml
Ammonium sulphate	40 Gm
Diluted sulphuric acid.................	30 ml

Heat the iron solution to the boiling point; add the ammonium sulphate, and, when this has dissolved, add the acid. Stir well. Set the mixture aside in a well covered vessel, in a cool place, for a day or two. Remove the crystals from the mother liquor, wash them hastily with a little cold water, dry them at once by pressing them gently between blotting paper, and put them, without delay, in a bottle, which must be tightly stoppered and kept in a cool place.

The crystals should be clean, well developed, and of a handsome violet color.

Reaction.

$$Fe_2(SO_4)_3+(H_4N)_2SO_4+24H_2O=2FeH_4N(SO_4)_2.12H_2O.$$

Notes. The sulphuric acid is added, because good crystals are best obtained when a slight excess of acid is present. If there is a deficiency of acid the crystals will be brownish, unclear, or very pale.

The color of the crystals of iron alum varies materially; it is sometimes bluish or purplish, sometimes violet, and sometimes a pale rose color. Crystals which are purplish violet may become almost devoid of color upon recrystallization from a water-solution unless the solution is rendered decidedly acid by the addition of H_2SO_4.

[Crystals of common alum grow in a saturated solution of iron alum. Large, clear, well defined crystals can thus be made which have a colorless center enveloped in a beautifully colored exterior. This is an interesting experiment for students to make.]

Description.—A violet colored salt, resembling alum in crystalline form, insoluble in alcohol, readily soluble in water, giving a brown solution.

Recrystallized Iron Alum.

Iron alum, when effloresced, discolored, or otherwise unsightly, may be recrystallized as follows:

Iron alum	100 parts
Water	300 parts
Diluted sulphuric acid	10 parts

Dissolve the salt in the acidified water with the aid of heat. filter the solution, and crystallize in the usual way.

LEAD ACETATE.

$Pb(C_2H_3O_2)_2.3H_2O=377.5.$

(*Sugar of Lead.*)

Litharge	5 parts
Acetic acid	8 parts
Water	4 parts

Mix the litharge, acid and water in a porcelain dish. Stir well. Let stand a day. Then heat over a water-bath until the oxide is dissolved. Filter. Evaporate until a pellicle forms and then set aside to cool and crystallize, adding first, if necessary, a little more acetic acid to impart to the solution a decidedly acid reaction. Collect the crystals, drain and dry them, immediately put the product in dry bottles, close these tightly, and keep them in a cool place.

Reaction.

$$PbO+2HC_2H_3O_2+2H_2O=Pb(C_2H_3O_2)_2.3H_2O.$$

Notes. The acetic acid dissolves the lead oxide without the aid of heat if sufficient time is allowed. But the solution is greatly hastened by the application of moderate heat. Too high a temperature would cause a loss of acetic acid.

Should the solution have a green color this shows the presence of copper which is sometimes contained in litharge. To remove this copper place a strip of pure lead in the solution; the copper is then deposited upon the lead and may be scraped off, after which the bright, clean lead is again placed in the solution. This must be repeated as long as any copper deposits upon the lead. When the liquid is free from copper, add a little more acetic acid, *if necessary,* to render the solution decidedly acid in its reaction on test-paper.

When too concentrated, solutions of lead acetate can not easily be filtered through paper. Should the solution be found too concentrated (by evaporation) to pass through the paper filter readily, dilute it with distilled water.

Should the crystals obtained contain iron acetate they will have a yellowish or rusty color. The salt must then be redissolved and recrystallized until colorless.

When perfectly clear and colorless crystals are formed the product is pure.

The mother liquor may be evaporated to one-half its volume, and, after adding a little more acetic acid, set aside for the formation of an additional crop of crystals.

Very large crystals can be obtained by the spontaneous evaporation of a solution saturated at the temperature of the laboratory; but very large crystals are not desirable for pharmaceutical uses. Smaller needle-shaped crystals are to be preferred, and may be obtained on cooling solutions saturated at an elevated temperature. Very small crystals are obtained by turbidation; but lead acetate in very small crystals is too liable to become contaminated with carbonate on exposure to the air.

This salt must be drained and dried as rapidly as possible, but without the aid of heat, or at a very moderate temperature, for the crystals are liable to dissolve in adhering mother liquor if wet, or to effloresce if dry.

When large amounts are prepared the crystals may well be drained on a muslin strainer and dried in an atmosphere of acetic acid produced by sprinkling a little of the acid about the drying salt. The product, if the quantity is not too large, may be most conveniently and safely dried by gently pressing it between blotting paper, changing the paper several times and as soon as moist, until the salt is dry.

Moist lead acetate rapidly absorbs carbonic acid from the air, if exposed, and forms insoluble carbonate. The surface of the crystals becomes white when covered with carbonate.

The salt also easily loses water of crystallization.

Lead acetate must therefore be quite dry before it is bottled, the containers must be dry, quite filled, tightly closed, and put in a cool place.

Description.—Colorless, clear, crystals of a faintly acetous odor, and sweetish, astringent, finally nauseous metallic taste. Efflorescent. Becomes whitish on exposure to air both from loss of water of crystallization and by the formation of carbonate due to the absorption of carbon dioxide from the air.

Soluble in 2.3 parts of water, and in 21 parts of alcohol, at 15°; in 1.5 parts of boiling water and in 1 part of boiling alcohol.

At 40° it loses its water of crystallization, which amounts to 14.25 per cent; but it loses a portion of its water even at 15° on exposure.

LEAD CARBONATE.

$2PbCO_3.Pb(OH)_2=770.5.$

Lead nitrate	10 parts
Sodium carbonate	9 parts

Dissolve the lead nitrate and the sodium carbonate each in 100 parts of water, and filter the solutions separately. Pour the lead salt solution into the sodium carbonate solution, stirring constantly. Wash the precipitate with hot water; collect and dry it.

Reaction.

$$3Pb(NO_3)_2+3Na_2CO_3+H_2O$$
$$=2PbCO_3.Pb(OH)_2+6NaNO_3+CO_2.$$

Description.—A heavy, white powder, insoluble in water, but soluble with effervescence in acetic or nitric acid. Odorless and tasteless.

LEAD IODIDE.

$PbI_2=457.5.$

Lead nitrate	1 part
Potassium iodide	1 part
Water, sufficient.	

Dissolve the lead nitrate in 8 parts of cold water, and the potassium iodide in 3 parts of cold water. Filter the solutions.

Add the lead solution to the solution of potassium iodide, with stirring. Decant the mother liquor and throw it away. Collect the precipitate on a filter and wash it with cold water. Dry it between bibulous paper.

Crystallization. If a crystalline product is desired, dissolve the washed and still wet precipitate by boiling it with two hundred times its weight of water, filter, and let the solution cool very slowly. The lead iodide obtained from 10 Gm of lead nitrate would require nearly three liters of boiling water for its solution. To obtain a small quantity of crystallized lead iodide as a specimen, boil the precipitate with 200 times its weight of water, filter while hot, and set the filtrate aside to cool slowly. The undissolved portion should be reserved separately.

Reaction. $Pb(NO_3)_2+2KI=2KNO_3+PbI_2$.

Description.—A heavy, bright-yellow powder; odorless and tasteless. Soluble in 2,000 parts of water at 15°, and in about 200 parts of boiling water.

LEAD NITRATE.

$Pb(NO_3)_2=329.5$.

Lead oxide, in fine powder...........	23 parts
Nitric acid (68%)....................	20 parts
Water	100 parts

Heat the oxide, acid and water together in a porcelain dish until the oxide has dissolved; filter the solution; acidify it by adding about 0.50 part of nitric acid; evaporate and crystallize.

Reaction. $PbO+2HNO_3=Pb(NO_3)_2+H_2O$.

Notes. If copper be present in the lead oxide the solution of lead nitrate will be bluish instead of colorless. Iron would make it yellowish. Copper may be removed by precipitation on metallic lead as described in the notes under plumbi acetas. Iron is removed by repeated recrystallizations.

Clear crystals cannot be obtained unless the solution of lead nitrate is decidedly acid; hence, after saturating the nitric acid with lead oxide, a small quantity of free nitric acid must be added to the solution before it is set aside to crystallize.

The solution must also be saturated only at the room temperature, and the crystals formed by spontaneous evaporation of the cold solution.

In the absence of free nitric acid the crystals obtained will be white or opaque; also when formed by cooling hot solutions.

The mother-liquor should be evaporated for more crystals.

Commercial impure lead nitrate may be purified by recrystallization from a water solution acidified with nitric acid, any copper or iron present being first removed as already described.

Description.—Colorless, transparent crystals, odorless, sweetish, astringent, taste finally metallic. Soluble in 2 parts of water at 15° and in 0.75 part of boiling water. Insoluble in alcohol. Reaction acid.

LEAD OXIDE.

$PbO = 221.5$.

Lead oxide is prepared on a large scale by oxidizing the metal at high temperatures in special furnaces. The purity of the product formed depends upon the purity of the lead used, and the degree of oxidation on the temperature and the access of air. The color also depends upon the temperature at the time of oxidation, and further upon the subsequent process of cooling. When slowly cooled the lead oxide is yellowish-red; but when rapidly cooled it is reddish-yellow.

Lead oxide, PbO, may be made by heating lead nitrate or lead carbonate until completely decomposed. The residue is the oxide. When pure lead nitrate is employed a very pure lead oxide is obtained if the heat be continued until red vapors cease to be given off and the residue acquires a constant weight. The nitrate should first be powdered, the heat gradually increased, and the powder stirred.

Should the residue be partly yellow and partly red, as it always will be if not thoroughly stirred or mixed, it is triturated until of perfectly uniform color.

Pure lead oxide is completely soluble in solution of normal acetate of lead.

LEAD PEROXIDE.

$PbO_2 = 237.5$.

Lead nitrate	5 parts
Chlorinated lime	8 parts
Water.	

Dissolve the lead salt in 30 parts of hot water. Triturate the chlorinated lime well with 60 parts of cold water and strain. Add the chlorinated lime solution in portions to the hot lead salt solution in a dish, stirring well. When about one-half of the solution of chlorinated lime has been added, heat the mixture until the light colored precipitate turns dark brown. Then filter about 10 ml of the liquid and test it by adding a little of the chlorinated lime solution and warming.

If a precipitate is formed, add more chlorinated lime solution to the mixture in the dish. Test again from time to time in the same way until no further precipitation is caused by the chlorinated lime. Then heat the mixture nearly to the boiling point, stirring well. Let settle, and wash the precipitate with boiling water, by decantation, until the washings are tastelss or free from chloride or nitrate. Collect and dry the peroxide.

Reaction.

$$Pb(NO_3)_2 + 2Ca(ClO)_2 + H_2O$$
$$= PbO_2 + CaCl_2 + Ca(NO_3)_2 + 2HCl + 2O.$$

Description.—A heavy, dark-brown, insoluble powder.

MAGNESIUM CARBONATE.

$4MgCO_3.Mg(OH)_2.aq.$

In pharmacy distinction is made between "heavy magnesium carbonate" and "light magnesium carbonate." The light magnesium carbonate differs from the heavy variety in these respects: the light carbonate is about three times as bulky; when examined under the microscope it is found to be partly amorphous, but containing numerous slender prismatic crystals, whereas the heavy variety is wholly amorphous and somewhat granular; the light magnesium carbonate yields less magnesium oxide on calcination.

These differences depend upon the methods employed in the preparation of the products. The working formulas given in the British Pharmacopœia are as follows:

Light Magnesium Carbonate.

Magnesium sulphate..................	25 parts
Sodium carbonate.....................	30 parts

Dissolve each salt in 200 parts of cold distilled water, filter, and mix the solutions *cold*. Boil the mixture in a porcelain dish for fifteen minutes. Transfer the precipitate to a wetted muslin strainer, and wash it with boiling distilled water until the washings cease to give a precipitate with test solution of barium chloride. Then let the precipitate drain, and dry it at not over 100° C.

Heavy Magnesium Carbonate.

Magnesium sulphate..................	25 parts
Sodium carbonate.....................	30 parts

Dissolve the salts, each in 50 parts of boiling distilled water, filter, and mix the two solutions while *hot*. Evaporate the mixture on a sand-bath to perfect dryness. Digest the residue for half an hour with 100 parts of boiling water, collect the precipitate on a wetted muslin strainer, and wash it repeatedly with distilled water until the washings cease to give a precipitate with test solution of barium chloride. Drain the product and dry it at not over 100° C.

Reaction. $4MgSO_4+4Na_2CO_3+5H_2O=$

$3MgCO_3.Mg(OH)_2.4H_2O+4Na_2SO_4+CO_2$; or perhaps

$5MgSO_4+5Na_2CO_3+6H_2O=4MgCO_3.Mg(OH)_2.5H_2O$
$+5Na_2SO_4+CO_2$.

Notes. It will be seen that the differences in manipulation are: the use of more dilute and cold solutions in making the light magnesium carbonate, and the precipitate is in that case simply heated for fifteen minutes with the mother liquor; while, in making the heavy magnesium carbonate the solutions are four times as concentrated, and mixed while hot, the mixture being then evaporated to perfect dryness on a sand-bath, after which the residue is freed from sodium sulphate by digestion and subsequent washing with boiling water.

When the precipitate is formed with cold solutions, it contains rather less magnesium than if prepared with hot solutions. When boiled in the liquid for a short time carbonic acid passes away; in preparing the light magnesium carbonate the heating should be discontinued as soon as the evolution of carbonic acid has commenced. When formed with hot, concentrated solutions, the mixture being boiled down to dryness, the precipitate contains less carbonic acid when formed, and loses more of that acid afterwards when exposed to the stronger heat.

Light magnesium carbonate contains a greater proportion of true carbonate ($MgCO_3$) and a correspondingly less proportion of hydroxide ($Mg(OH)_2$). The quantity of water in the product seems also to vary.

Description.—Light, white, friable masses, or a light, white powder; odorless; produces a slightly earthy feel in the mouth. Practically insoluble in pure water, although it imparts to it a slightly alkaline reaction. Insoluble in alcohol.

The foregoing description applies to the light variety. The heavy carbonate is a powder only one-third as bulky.

MAGNESIUM OXIDE.

MAGNESIUM OXIDE; U. S. P. LIGHT MAGNESIA. LIGHT MAGNESIUM OXIDE.

(Calcined Magnesia.)

$MgO=40.2$.

Light magnesium carbonate.

Rub it through a No. 60 sieve. Heat it in a capacious unglazed earthenware dish, with constant stirring, on a sand-bath, until a sample removed from the center, cooled, mixed with a little water, and then dropped into diluted sulphuric acid, gives but a very slight effervescence.

Reaction. $(MgCO_3)_4.Mg(OH)_2.(H_2O)_5$ when strongly heated is decomposed into $5MgO+6H_2O+4CO_2$.

Notes. The magnesium carbonate being very light, a great deal of it rises from the dish like dust and is scattered about.

In operating upon larger quantities a bright, clean iron pot may be used, placed immediately over the fire and heated gradually to dull redness.

If heated too strongly or too long the magnesia becomes "dead-burnt," so that it does not become "hydrated" and form a gelatinous mass of hydroxide when mixed with water. This failure of the oxide to react with water results whenever the carbonate is heated until all of it is completely decomposed so that no effervescence whatever occurs when the product is tested with diluted sulphuric acid. On the other hand, the product may, if not heated long enough, contain much basic anhydrous carbonate, although it gelatinizes with water; in such a case it does not retain the property of becoming converted into gelatinous hydroxide. A *slight* effervescence with diluted sulphuric acid is, therefore, allowable; but considerable effervescence with diluted acid should condemn the product.

Magnesia must be kept in small, filled, tightly-corked bottles, the corks dipped in melted paraffin to effectively exclude air so as to prevent the absorption of CO_2 and the consequent formation of carbonate. Containers capable of holding about 50 Gm of magnesia are perhaps the most suitable in size.

Test. When 1 Gm of magnesia ("light magnesia") is thoroughly stirred with 15 ml of water in a beaker and the mixture allowed to stand in the beaker for an hour, a semi-translucent gelatinous mass of magnesium hydroxide should be formed, which is firm enough to remain in the beaker when the latter is inverted. A magnesia which does not possess the quality to thus form hydroxide is not to be used medicinally, being inferior.

Description.—A white, very light and extremely fine powder; odorless and tasteless, but producing in the mouth the sensation usually produced by dry, absorbent, tasteless, earthy substances. Insoluble in alcohol, and practically insoluble in water, although when moistened it exhibits a slightly alkaline reaction on litmus paper.

"Light magnesia" is about three times as bulky, weight for weight, as "heavy magnesia."

Heavy Magnesia.

(Heavy Magnesium Oxide.)

$MgO=40.2$.

A white, very fine powder, about three times as dense as the light magnesia, but otherwise of similar physical properties. It also conforms to the reactions and tests given by the Pharmacopœia for "Magnesia," but does not form a gelatinous hydroxide with water, or does so only with great difficulty.

MAGNESIUM SULPHATE.

(Epsom Salt.)

$MgH_2SO_5.6H_2O=246.2$.

Magnesite, in powder.................	20 parts
Sulphuric acid (92.5% of H_2SO_4)......	25 parts
Water	70 parts

Mix the magnesite in a porcelain dish with 40 parts of water. Then add about 2 parts of the sulphuric acid, and stir. When effervescence has ceased add another portion of the acid, and again wait until the reaction has subsided. Continue adding sulphuric acid in portions in the same manner until about two-thirds of it has been used. Then add about 20 parts of water, and heat the mixture to about 90°. Add the remainder of the acid, in portions, as before; then add the rest of the water, and heat the whole at about 90° for two hours, stirring frequently. Add enough magnesium carbonate to render the solution neutral to litmus paper. Set the liquid aside to cool and settle. Filter. Evaporate the filtrate until it has about 1.33 sp. w. while still hot, and then set it aside to crystallize. Drain the crystals and dry them at about 25°.

Reaction. $MgCO_3+H_2SO_4=MgSO_4+H_2O+CO_2$

Description.—Small, colorless, acicular crystals; odorless; taste cooling, saline, bitter. Slightly efflorescent in dry air. Soluble in 1.5 parts of water at 15° and in 0.7 part of boiling water. Insoluble in alcohol.

MANGANESE PHOSPHATE.

$Mn_3(PO_4)_2.4H_2O=427$.

Manganous sulphate	10 parts
Sodium phosphate	11 parts

Dissolve the salts, separately, each in 80 parts of hot water, filter, and let the solutions cool. Mix them. Wash the precipitate with water until the washings cease to give any reaction for sulphate. Dry the product with the aid of gentle heat, avoiding unnecessary exposure to the air.

Reaction.

$$3(MnSO_4.4H_2O)+2(Na_2HPO_4.12H_2O)$$
$$=Mn_3(PO_4)_2.4H_2O+2(Na_2SO_4.10H_2O)+H_2SO_4+12H_2O.$$

Notes. As will be seen, the reaction is analogous to that occurring in the preparation of ferroso-ferric phosphate. Loss of product is sustained by reason of the solubility of manganous phosphate in the sulphuric acid formed. The free acid may be carefully neutralized by the addition of sodium bicarbonate to obviate the loss.

Description.—An almost white (slightly pinkish) powder, odorless and tasteless; insoluble in water and in alcohol.

MERCURIC BENZOATE.

$Hg(C_6H_5CO_2)_2=440.5.$

Freshly precipitated mercuric oxide from 27 parts of mercuric chloride is mixed with 22.5 parts of artificial benzoic acid (from toluol) and the mixture digested at a temperature near the boiling point (at full water-bath heat) until the yellow color of the mercuric oxide has given place to the yellowish-white color of the mercuric benzoate formed. The product forms voluminous masses floating in the liquid.

Let the liquid cool to 50°C., transfer the whole mixture to a cloth strainer or a paper filter, according to the quantity operated upon, and wash the benzoate with water of the temperature of from 50° to 60° C.

Dissolve the salt in boiling water and let the solution cool so that the product may be obtained in crystals. Dry these at from 40° to 50° C.

Description.—Mercuric benzoate thus prepared is in colorless, glistening crystal needles, of a somewhat metallic taste.

Gives an acid reaction on moist blue litmus paper. Nearly insoluble in cold water, but freely soluble in boiling water. Readily soluble in a water solution of sodium chloride, and this solution has been used for hypodermic injection. Soluble in alcohol but with partial decomposition. It contains about 45.2 per cent of Hg.

MERCURIC CHLORIDE.

(Corrosive Sublimate. Corrosive Chloride of Mercury.)

$HgCl_2=269.3$.

Mercuric sulphate	20 parts
Sodium chloride	15 parts
Manganese dioxide	1 part

Mix the dried and powdered materials and triturate the mixture thoroughly until a uniform and very fine powder is obtained. Put this into a suitable vessel and heat in a sand-bath until the mercuric chloride has been sublimed.

Reaction. $HgSO_4+2NaCl=Na_2SO_4+HgCl_2$.

Notes. The materials must be thoroughly dry and reduced to very fine powder. The manganese dioxide should be strongly heated after having been powdered and before it is mixed with the other substances.

On a small scale a retort may be used having the neck cut off rather short so as to leave a wide opening. The retort should be buried in the dry sand up to the neck. Heat is then applied gradually. If the materials were not perfectly dry water vapor will be formed, and it is then necessary to heat gently at first until the moisture shall have been expelled, and even the neck of the retort should be warmed sufficiently to insure the expulsion of all water vapor. The iron dish employed for the sand-bath is then heated more and more strongly until sublimation is effected, the end of the neck of the retort being capped with a wide-mouthed bottle. The heat is continued until the sublimation is completed, care being taken not to permit the neck of the retort to become choked up. Towards the close of the process the heat is raised almost to the fusing point of the mercuric chloride in order to obtain a sublimate of more decidedly developed crystalline structure. The retort is finally allowed to cool slowly, after which the sublimate may be readily removed from the short neck.

In order to obviate the danger of inhaling any vapor of mercuric chloride it is necessary that the whole operation be carried out in a hood with a good draught. The product must not be carelessly handled, being extremely poisonous. If it is desired to powder corrosive sublimate, it should be kept dampened with alcohol during the process of trituration, to prevent the rising of dust.

Should the mercuric sulphate employed contain any mercurous sulphate a corresponding quantity of mercurous chloride must be produced. To prevent this the manganese dioxide is added. The rationale of its action is shown in the following equation:

$$Hg_2SO_4+MnO_2+2NaCl=Na_2SO_4+2HgO+MnCl_2.$$

Without the addition of manganese dioxide it is scarcely possible to obtain a corrosive sublimate that does not contain calomel to such an extent as to produce a very unclear solution in water. But the sublimed mercuric chloride may be dissolved in diluted alcohol and crystallized from the filtered solution.

Another Method.

Mercuric oxide	9 parts
Hydrochloric acid (32% of HCl)	10 parts
Distilled water	500 parts

Heat together until the oxide is completely dissolved. Filter the hot solution, and evaporate it to dryness. Crystallize the product.

Reaction. $HgO+2HCl=HgCl_2+H_2O$.

Notes. The product obtained by this method may be sublimed to obtain it in the form of crystals or crystalline masses, or it may be crystallized in needles from a solution made with warm diluted alcohol, or with water.

Solutions of mercuric chloride for certain purposes may be readily made of definite strength from mercuric oxide, hydrochloric acid and water in such proportions as may be required. The hydrochloric acid is prescribed in slight excess.

Description.—Heavy, transparent, colorless, odorless crystals or crystalline masses, having an acrid, nauseous, persistent, metallic taste. It is extremely poisonous, and should, therefore, not be tasted except in the most cautious manner.

Obtained by sublimation on a large scale, or heated at or near its fusing point (about 265°) and then very slowly cooled, it forms large, transparent, colorless crystals. When crystallized from solutions it is obtained in the form of long needles which are somewhat opaque. Soluble in 16 parts of water, and in 3 parts of alcohol, at 15°; in 2 parts of boiling water, and in 1.2 parts of boiling alcohol; also in 4 parts of ether, and in 14 parts of glycerin.

Mercuric chloride dissolves so slowly in water that in making solutions of it the most advantageous procedure is to employ powdered chloride and hot water.

MERCURIC BROMIDE.

$HgBr_2=356.5$.

Mercury	10	parts
Bromine	8.5	parts
Water	350	Cc

Place the metal in a flask; add 50 Cc of the water; then the bromine. Shake the mixture until the mercury is converted into a white pulverulent mass. Then add the remainder of the water, boil for a few minutes, filter and set aside to crystallize.

Reaction. $Hg+2Br=HgBr_2$.

Notes. The action is at first slow, but when some $HgBr_2$ has been formed the bromine dissolves more freely and the reaction goes on rapidly.

If necessary, a little more bromine should be added to complete the solution of the last of the mercury.

Description.—A white, odorless, crystalline solid; soluble in about 200 parts of water at the ordinary temperature. Much more soluble in boiling water.

MERCURIC OLEATE.

$Hg(C_{18}H_{33}O_2)_2=760.5$.

Mercuric oxide	30 Gm
Nitric acid (1.42 sp. gr.)............	26 Gm
Solution of potassium oleate.........	1,500 ml

Mix the oxide, the acid, and 200 ml of distilled water, in a beaker or an evaporating dish, and heat the mixture until the oxide dissolves, adding a few drops more of nitric acid, *if necessary.* Dilute the solution of mercuric nitrate thus formed with three liters of water. Add 1,500 ml of solution of potassium oleate. Warm the mixture and wash the separated mercuric oleate twice with warm distilled water, using about one liter each time. Subject the oleate to a low temperature in a mortar, and then press out of it all the water by means of a pestle.

Reaction.

$Hg(NO_3)_2+2KC_{18}H_{33}O_2=Hg(C_{18}H_{33}O_2)_2+2KNO_3$.

The oleate of mercury U. S. P. is made from oleic acid, water and 25 Gm of mercuric oxide; the finished product to weigh 100 Gm.

Notes. Mercuric chloride cannot be employed in place of the nitrate, nor can sodium oleate be used instead of the potassium oleate, as the washing then becomes very difficult.

The yield is about 100 Gm or a little more. The product is a soft, yellow or reddish-yellow solid, and contains 28.4 per cent of mercuric oxide.

Mercuric oleate is very sensitive to heat, decomposing with the separation of finely divided, dark-colored, metallic mercury. Precipitated oleate of mercury containing no free oleic acid keeps better than solutions of mercuric oleate in free oleic acid.

Solution of potassium oleate is prepared as follows:

Potassium bicarbonate	37.5	Gm
Oleic acid..........................	105	Gm

Add the potassium carbonate gradually to 1,500 Cc of boiling water. When effervescence has ceased, add the oleic acid, stir well, and continue heating the mixture gently, adding more water if necessary, until saponification is complete. Then dilute the solution with enough water to make it measure 2 liters.

MERCURIC OXIDE; RED.

$HgO=214.5$.

Dissolve 8 parts of mercury in 9 parts of official nitric acid previously diluted with 4 parts of water. Evaporate the solution to dryness. To the residue add another 8 parts of mercury, and triturate in a mortar until metallic globules are no longer visible and the mixture appears to be uniform. Heat this mixture in a porcelain dish over a sand-bath, stirring frequently, until acid vapors are no longer evolved.

Reactions.

First, $3Hg+8HNO_3=3Hg(NO_3)_2+4H_2O+2NO$.

Second, $Hg(NO_3)_2+Hg=Hg_2(NO_3)_2$.

Third, $Hg_2(NO_3)_2$, when heated, decomposes into $2HgO+N_2O_4$.

Notes. The nitrate is decomposed by heat, red fumes (nitrogen tetroxide) being evolved. When all the nitrate has been thus decomposed, the acid vapors cease, and the residue is mercuric oxide. Care must be taken to continue the heating until not a trace of nitrate remains. Mercuric oxide when strongly heated becomes very dark, but the red color returns on cooling. If the heat is too high (over 360° C.) the oxide decomposes into its elements, which may be discovered by holding a piece of filter-paper over the heated mass, when metallic mercury will condense on the paper.

It is sometimes directed to digest the residue after cooling, with distilled water to which a little solution of soda has been added, the object being to remove any remaining nitrate. In this case the oxide must afterwards be thoroughly washed, and dried at a gentle heat.

Description.—A brick-red, insoluble powder. Must not produce red vapors when heated. It is completely soluble in HCl, producing $HgCl_2$.

PRECIPITATED MERCUROUS CHLORIDE.

$HgCl=233.7.$

(Precipitated Calomel.)

Mercury	175 parts
Nitric acid, 68%	150 parts

Distilled water, sodium chloride, and hydrochloric acid.

Put the mercury in a tared flask; add 100 parts nitric acid and 150 parts water. Heat the flask moderately on a sand-bath, covering the mouth of the flask with a watch-glass, and continue the heat without interruption for one hour after red fumes have ceased to be evolved, or until the liquid begins to acquire a yellowish color and to become slightly turbid. Pour the solution of mercurous nitrate off from the undissolved mercury.

Mix 50 parts of nitric acid with 1,000 parts of boiling distilled water, and add this mixture to the solution of mercurous nitrate while still hot. Let the mixture cool.

Test the liquid to ascertain whether or not it will bear dilution with an equal volume of cold distilled water without becoming turbid. To perform this test mix about 5 ml of the solution with 5 ml of distilled water. Should the mixture remain clear, return it to the whole quantity of the mercury solution. Should it become turbid, add a sufficient quantity of nitric acid to the whole solution to acidify it so that dilution with twice its volume of water without precipitation is rendered assured.

Ascertain the quantity of mercury contained in the solution by deducting from the quantity originally taken the weight of the undissolved portion remaining in the flask. [That undissolved mercury must be washed and dried before weighing it.]

Take a quantity of sodium chloride equal to the weight of the dissolved mercury; dissolve that sodium chloride in 20 times its weight of distilled water, and add to this solution 5 per cent of its weight of hydrochloric acid. Then pour the sodium chloride solution gradually into the mercury solution, stirring constantly.

Wash the precipitate with cold distilled water, by affusion and decantation, until the washings no longer give an acid reaction on test-paper.

Dry the product at a temperature not exceeding 40° C.

Reactions. $3Hg+4HNO_3=3HgNO_3+2H_2O+NO$, and subsequently $HgNO_3+NaCl=HgCl+NaNO_3$.

Notes. It is necessary that the mercury solution should contain enough nitric acid to remain clear when diluted with its own volume of water, and it is to be tested to make sure of that fact before the solution of sodium chloride is added, because if the mercury solution should contain too little nitric acid the precipitate formed upon dilution with water is a basic mercurous nitrate and this would then be precipitated by the solution of sodium chloride together with the calomel. But if the solution of mercurous nitrate will bear dilution with its own volume of water without becoming turbid, it may be safely mixed with more than its own volume of sodium chloride solution, gradually added, without any danger of the precipitation of basic mercurous nitrate together with the mercurous chloride.

The large dilution of the liquids is necessary to prevent the formation of mercuric salts by the nitric acid.

Precipitated calomel is much bulkier than sublimed calomel, and is extremely finely divided.

It should be tested for nitrate, from which it must be quite free.

Description.—Precipitated calomel is a finely divided, white and tasteless powder. It is bulkier and finer than sublimed calomel.

POTASSIUM ACETATE.

$KC_2H_3O_2=98$.

Acetic acid	5 parts
Potassium bicarbonate	3 parts

Add the potassium bicarbonate gradually to the acid; filter; evaporate over a sand-bath to dryness, stirring constantly, adding a little acetic acid if necessary to keep it in decided excess to the end.

Must be kept in tightly corked bottles.

Reaction. $KHCO_3+HC_2H_3O_2=KC_2H_3O_2+CO_2+H_2O$.

Notes. Theoretically the proportions given in the foregoing formula leave the acetic acid slightly in excess; but this excess of acid will be driven off during the evaporation, and more must be added from time to time as may be required in order to maintain a slightly acid reaction.

The temperature during the evaporation should be 120° to 140° C.

POTASSIUM BROMIDE.

$KBr=118$.

Iron wire, cut	5 parts
Bromine	12 parts
Potassium bicarbonate	15 parts
Distilled water.	

Put the iron together with 40 parts of distilled water in a flask. Add 9 parts of bromine, in small proportions at a time, shaking the flask after each addition. Heat the flask and contents if necessary to cause the reaction to be completed—i. e., until all odor of bromine ceases and a green solution of ferrous bromide results. Filter the solution. Add the remainder of the bromine to the filtrate and shake well.

Put the potassium bicarbonate in a porcelain dish with 50 parts of distilled water, heat until effervescence ceases, and then raise the heat to boiling.

Add slowly to the boiling solution of potassium carbonate the solution of bromide of iron. Boil the mixture for fifteen minutes. The solution should now have a slightly alkaline reaction. If not, add more potassium carbonate or bicarbonate until the reaction is alkaline.

Let the precipitate subside. Filter the solution. Wash the precipitate on the filter with hot distilled water and add the washings to the filtrate. Evaporate the solution to the requisite degree of concentration and set it aside to crystallize. Drain the crystals in a funnel. Then spread the salt upon bibulous paper and dry it with the aid of moderate heat.

Additional crystals may be obtained from the mother liquor in the usual way.

Instead of crystallizing the salt the solution may be evaporated to dryness and the bromide granulated.

Reaction.

$Fe+2Br=FeBr_2$; then $3FeBr_2+Br_2=FeBr_2.2FeBr_3$;

and, finally, $FeBr_2.2FeBr_3+4K_2CO_3$
$=8KBr+FeO.Fe_2O_3+4CO_2$.

Notes. Ferroso-ferric bromide gives better results than ferrous bromide because the iron is more readily and completely precipitated in the form of ferroso-ferric oxide.

Potassium bromide crystallizes best from a slightly alkaline solution. But if the salt is to be recovered in a granular condition by evaporation to dryness with constant stirring, then the solution should be neutral. It is, however, necessary to render the solution faintly alkaline for the purpose of precipitating all the iron. The liquid can afterwards be made neutral again by adding enough hydrobromic acid, which, for this purpose, may well be made from potassium bromide and tartaric acid.

To improve the appearance of potassium bromide the crystals may, after drying, be heated for a few minutes at a temperature of 80° to 100°.

Description.—Colorless or white cubical crystals, or a white granular salt; odorless, and of saline, pungent taste. Soluble at 15° in about 1.6 parts of water and in 200 parts of alcohol; in less than its own weight of boiling water, and in 16 parts of boiling alcohol. Soluble in four times its weight of glycerin. The water-solution must have a neutral reaction on test-paper, or only a very faintly alkaline reaction.

POTASSIUM CARBONATE.

["Salts of Tartar."]

$K_2CO_3=138$.

Potassium bicarbonate, any desired quantity.

Put the salt into a porcelain dish, cover it well with distilled water, heat gradually and continue the heat until effervescence ceases and the liquid boils. Then evaporate to dryness, stirring constantly. Put the product at once into a perfectly dry bottle and close this tightly.

Reaction. $2KHCO_3=K_2CO_3+H_2O+CO_2$.

Notes. To obtain a perfect product the potassium bicarbonate used must be perfectly clean and pure. If it makes an unclear solution in water, the bicarbonate should all be dissolved in about five or six times its weight of water and the solution filtered before it is heated to decompose it into normal carbonate, water and carbon dioxide.

The product must be perfectly dry and white.

Description.—Potassium carbonate, sufficiently pure to fulfill the requirements of the Pharmacopœia, is a white granular powder, odorless, of strongly alkaline taste. It is very deliquescent. Soluble in 1.1 parts of water at 15°, and in 0.65 part of boiling water. Insoluble in alcohol.

TURBIDATED POTASSIUM CHLORATE.

$KClO_3=122.4$.

Potassium chlorate	1 part
Boiling distilled water	5 parts

Dissolve and filter. Stir the filtrate constantly until cold. Collect the crystals on a filter, let drain, and then dry the product.

Notes. Commercial potassium chlorate usually contains potassium chloride and calcium chloride. These impurities are much more readily water-soluble than potassium chlorate. Hence the turbidation of the salt results also in purification, for the chlorides remain in the mother liquor.

When cooled to 15° C. the mother liquor still retains about 6 per cent of its weight of chlorate, or 30 per cent of the amount of salt operated upon. This must me recovered as far as practicable, which can be effected by evaporating the mother-liquor by water-bath heat to about one-third its bulk, stirring until cold, and collecting, draining and drying the "second crop" of small crystals in the same manner as the first.

Description.—Turbidated potassium chlorate is a glistening, white, crystalline powder, odorless, of a cooling saline taste.

Soluble in 16.7 parts of water at 15°; in about 3 parts of water at 75°, and in 1.7 parts of boiling water. Nearly insoluble in alcohol. Explosive with certain readily oxidized substances, especially when heated with them, or mixed with reducing agents, by trituration, or by concussion. A powerful oxidizing agent.

POTASSIUM CHLORIDE.

$KCl=74.4$.

Potassium bicarbonate	100 parts
Diluted hydrochloric acid	364 parts
Distilled water	100 parts

Heat the potassium bicarbonate in a porcelain dish with the water until converted into normal carbonate. Then add this gradually to the hydrochloric acid, stirring well. When the solution is neutral to test-paper, filter it, and evaporate the filtrate to crystallization or granulation, as may be desired.

Reaction. $K_2CO_3+2HCl=2KCl+H_2O+CO_2$.

Description.—Colorless translucent crystals, or a white granular salt; odorless and of a pure saline taste. Soluble at 15° in about three times its weight of water, and in less than one-half its weight of boiling water. Insoluble in alcohol.

POTASSIUM IODATE.

$KIO_3=213$.

Potassium permanganate	2 parts
Potassium iodide	1 part
Water,	
Alcohol,	
Acetic Acid.	

Dissolve the permanganate in 50 parts of hot water, add the iodide dissolved in 2 parts of water, heat the mixture over a water-bath for about twenty minutes or half an hour, adding alcohol drop by drop at the end until the liquid is decolorized, and then filter. Wash the precipitate on the filter and add the washings to the filtrate. To the filtrate add enough acetic acid to render the reaction on test-paper distinctly acid. Evaporate the liquid to about 1.5 parts; let it cool; separate the crystals from the mother-liquor, wash the product with alcohol and dry it.

Reaction. $3KI+6KMnO_4+3H_2O=6MnO_2+3KIO_3+6KOH$.

Notes. The acidification with acetic acid is necessary to prevent the product from having an alkaline reaction due to adhering alkali, which it is extremely difficult to wash away.

Description.—A white, granular, crystalline salt of perfectly neutral reaction to test-paper.

POTASSIUM IODIDE.

$KI=165$.

Solution of potassium hydroxide.....	1,000 ml
Iodine.....................	100 Gm, or sufficient
Wood charcoal, in fine powder..........	15 Gm
Boiling distilled water, sufficient.	

Put the solution of potassium hydroxide in a flask, add the iodine, a little at a time, with constant agitation, until the solution becomes permanently brown. Evaporate to dryness. Powder the residue, and mix it well with the charcoal. Throw the mixture into a red-hot iron crucible, and when the whole is in a fused condition pour it out to cool, dissolve it in 200 ml of boiling distilled water, filter, and evaporate until a pellicle forms. Then set aside to cool and crystallize. Drain the crystals and dry them in a warm place. The remainder of the salt may be obtained from the mother liquor by evaporation to dryness, stirring constantly.

Reaction. $3I_2+6KOH=5KI+KIO_3+3H_2O$; and then,

$2KIO_3+3C=2KI+3CO_2$.

Notes. The red brown liquid formed by dissolving iodine in solution of potassium hydroxide contains potassium iodide and potassium iodate, as seen from the above equation. By heating the salt mass obtained on evaporation of this liquid to dryness, the iodate is reduced to iodide; but the heat necessary for this purpose is so high that loss of iodine ensues, and the product becomes alkaline. To prevent this, charcoal is added, which makes the reduction practicable at a much lower temperature.

The crystallization of potassium iodide is somewhat difficult. It is best to continue the evaporation until a sample of the liquid, when removed from the vessel, crystallizes on cooling. The liquid should then be left in a warm place (50° to 60°), where the evaporation may continue spontaneously in order that large crystals may be obtained. At a lower temperature the iodide ascends the sides of the vessel and creeps over the edges.

When perfectly pure, potassium iodide is difficult to obtain in colorless crystals; it undergoes partial decomposition with liberation of enough iodine to discolor the product. To prevent this discoloration the liquid is usually rendered slightly alkaline before being set aside for crystallization. The presence of potassium carbonate not only facilitates the formation of crystals, but also renders the product whiter. The pharmacopœial tests permit a slight alkalinity (less than 0.1 per cent). Crystals of perfectly pure potassium iodide are usually not quite white, but may be made so by drying at 120° to 125° C.

Second Method.

Iron wire, cut	6 parts
Iodine	20 parts
Potassium bicarbonate	16 parts
Distilled water, sufficient.	

Digest the iron and 15 parts of the iodine with 50 parts of water in a flask until all odor of iodine ceases and a green solution of ferrous iodide results. Filter this, and in the filtrate dissolve the remainder of the iodine.

Put the potassium bicarbonate into a dish with 80 parts of distilled water, heat until effervescence ceases, and then raise the heat to boiling. Add slowly to this boiling hot solution the filtered solution of the iodides of iron. Boil the mixture for fifteen minutes. (If all the iron has not been precipitated from the solution, add a little more potassium bicarbonate.) Filter, evaporate, and crystallize.

Reaction. $Fe+I_2=FeI_2$; then $3FeI_2+I_2=FeI_2.2FeI_3$; finally

$FeI_2.2FeI_3+4K_2CO_3=8KI+FeO.Fe_2O_3+4CO_2$.

Notes. As seen from the equation, a solution of ferrous iodide is first made. This can be at once treated with potassium carbonate to obtain the potassium iodide:

$FeI_2+K_2CO_3=2KI+FeCO_3$.

But in this case the iron is separated with greater difficulty, being first thrown down as ferrous carbonate, which at once begins to lose carbonic acid, and at the same time oxidizes so that the precipitate gradually changes from light gray to blue, yellowish brown, and finally to brown ferric hydroxide $Fe(OH)_3$. All of the iron does not separate at once, however, so that during the subsequent evaporation of the filtered solution of iodide of potassium a considerable quantity of flocculent deposit of ferric hydroxide makes its appearance and must be filtered out.

This difficulty may be remedied to a great extent by converting the ferrous iodide into ferrico-ferrous iodide by adding the required additional quantity of iodine. The precipitate will then, after the boiling, be black magnetic oxide of iron, which is very dense and, therefore, more easily separated than ferric hydroxide.

When the solution of iodide of iron has all been added to the hot solution of potassium carbonate, the liquid should have a neutral or faintly alkaline reaction. Should it instead be acid, add more potassium bicarbonate. If too alkaline add some hydriodic acid prepared from tartaric acid and KI.

POTASSIUM OXALATE; NORMAL.

$K_2C_2O_4.2H_2O=202.$

Oxalic acid	63 parts
Potassium bicarbonate	100 parts
Distilled water	500 parts

Dissolve the oxalic acid in the water by the aid of heat; then add the potassium bicarbonate. Neutralize perfectly. Evaporate and crystallize.

Reaction. $2KHCO_3+H_2C_2O_4=K_2C_2O_4+2H_2O+2CO_2.$

Description.—Transparent prisms or pyramids, easily soluble in water.

POTASSIUM-SODIUM TARTRATE.

Rochelle Salt.

$KNaC_4H_4O_6.4H_2O=282.$

Potassium bitartrate	5 parts
Sodium carbonate	4 parts
Distilled water	20 parts

Heat the water to boiling in a porcelain dish; add the sodium carbonate, and when that is dissolved, gradually add the cream of tartar, and continue heating until all is dissolved. Set the solution aside for a day or two. Then filter, evaporate the filtrate to nine parts, or until a pellicle forms, and then set it aside to crystallize.

Reaction.

$2KHC_4H_4O_6+Na_2CO_3=2KNaC_4H_4O_6+H_2O+CO_2.$

Notes. A slight excess of sodium carbonate is prescribed, because the salt crystallizes best from a slightly alkaline solution. Any excess of sodium carbonate will remain in the mother liquor. Large crystals are easily obtained. The crystals should be hastily rinsed with a little cold distilled water. The mother liquor is usually colored, more so the farther the evaporation is carried.

The direction to allow the solution to stand some time before filtering and evaporating to crystallization, has for its object the deposition of any calcium tartrate and carbonate.

The crystals must be dried without the aid of heat.

After several crystallizations, the mother liquor becomes colored. It may be decolorized with animal charcoal if the quantity operated upon justifies it. The tartaric acid in the last mother liquor may be recovered in the form of cream of tartar by precipitation with hydrochloric acid.

Description.—Colorless, transparent prisms, or a white powder; odorless; taste cooling, saline. Slightly efflorescent in warm, dry air. Soluble in 1.4 parts of water at 15°, and in less than its own weight of boiling water. Practically insoluble in alcohol. Neutral to litmus paper.

POTASSIUM SULPHATE.

$K_2SO_4=174$.

Diluted sulphuric acid................	100 parts
Potassium bicarbonate	20 parts

Place the acid in a porcelain dish and neutralize it with the potassium bicarbonate, gradually added, stirring after each addition until effervescence has ceased before adding more. Try the solution with test-paper, and add more potassium bicarbonate if necessary to render the reaction quite neutral. Filter the solution and evaporate to crystallization.

Reaction. $2KHCO_3+H_2SO_4=K_2SO_4+2H_2O+2CO_2$.

Description.—Colorless, odorless crystals of a somewhat bitterish saline taste. Soluble in 9.5 parts of water at 15°, and in 4 parts of boiling water. Insoluble in alcohol.

SODIUM ACETATE.

$NaC_2H_3O_2.3H_2O=136.$

Acetic acid (36%)	100 parts
Sodium carbonate	85 parts
Water	100 parts

Put the acetic acid and water in a porcelain dish. Add the coarsely powdered or crushed sodium carbonate in small portions, stirring after each addition until effervescence ceases before adding another portion. When all of the sodium carbonate has been added heat the liquid to the boiling point. Then add.

Sodium carbonate	2 or 3 parts

as may be required to render the solution quite neutral to test-paper, or only faintly acid. Filter. Evaporate the filtrate until a test-portion when cooled gives crystals. Then let the liquid stand until cold. Collect, drain, and dry the crystals.

Reaction. $Na_2CO_3+2HC_2H_3O_2=2NaC_2H_3O_2+H_2O+CO_2.$

Notes. When the solution is to be tested as to its reaction on test-paper the portion taken for this test should be first diluted with about three times its volume of water. Should the liquid be found to have an alkaline reaction more acetic acid must be added (and the whole well mixed) until the reaction is slightly acid.

The crystals must be dried at the ordinary temperature.

When acetic acid containing empyreumatic products is used, their odor comes out on neutralizing the acid. The acetate should then be carefully heated to complete fusion at a temperature of about 300° (and not exceeding 310°) for half an hour when the empyreumatic substances will be decomposed. The salt is then recrystallized.

Description.—Colorless, transparent crystals; odorless; taste saline, cooling. Efflorescent in warm dry air. Soluble in 1.4 parts of water and in 30 parts of alcohol at 15°; in half its weight of boiling water, and in twice its weight of boiling alcohol. The salt becomes liquid at 60°, and anhydrous at 123°. It decomposes at 315° C. Its water-solution (5%) turns red litmus paper blue, but should not redden phenolphtalein paper. If it reddens phenolphtalein it probably contains sodium carbonate.

SODIUM BENZOATE.

$NaC_7H_5O_2=144.$

Benzoic acid	40 parts
Sodium bicarbonate	28 parts
Boiling water	80 parts

Mix the benzoic acid with the hot water, stir well, neutralize perfectly by adding the sodium bicarbonate, being careful to observe the reaction on litmus paper after effervescence has ceased. Filter. Evaporate to 55 parts; remove it from the source of heat and stir until cold. During the evaporation it is necessary to scrape down the benzoate from the sides of the capsule.

Reaction. $2HC_7H_5O_2+Na_2CO_3=2Na_2C_7H_5O_2+H_2O+CO_2.$

Description.—A white, semi-crystalline or amorphous powder, efflorescent on exposure to air, odorless or having a faint odor of benzoin, of a sweetly astringent taste free from bitterness, and having a neutral reaction. Soluble in 1.8 parts of water, and in 45 parts of alcohol at 15° C.; in 1.3 parts of boiling water, and in 20 parts of boiling alcohol.

SODIUM CHLORATE.

$NaClO_3=106.25.$

Prepared from calcium hydroxide and sodium chloride with chlorine, or from chlorinated lime and sodium chloride, by a process analogous to that for preparing potassium chlorate.

It may also be made as follows:

Tartaric acid	117 parts
Sodium carbonate	110 parts
Potassium chlorate	96 parts

Dissolve the acid and the carbonate in 1,000 parts of hot water. To the hot solution add the potassium chlorate. Potassium bitartrate separates, and the solution contains the sodium chlorate together with some potassium bitartrate, which deposits during the evaporation to crystallization, the last being gotten rid of by crystallizing it out from the saturated solution of the sodium chlorate.

Description.—Colorless transparent crystals, or a white crystalline powder; odorless; taste saline, cooling. Soluble at 15° in 1.1 parts of water, and in about 100 parts of alcohol; in half its weight of boiling water and in 40 parts of boiling alcohol; in 5 parts of glycerin. The water-solution is neutral to test-paper. Like potassium chlorate the sodium chlorate must be handled with care, and not triturated with reducing agents or subjected to concussion.

SODIUM CHLORIDE.

$NaCl=58.4.$

Diluted hydrochloric acid	5 parts
Sodium carbonate	2 parts

Add the sodium carbonate gradually to the diluted acid. Make the solution quite neutral to litmus paper. Filter. Evaporate to dryness, or to crystallization, as may be desired.

Reaction. $Na_2CO_3+2HCl=2NaCl+H_2O+CO_2$.

Purification of Common Salt.

Common salt	1	kilogram
Water	3½	liters
Lime	12	Gm
Barium chloride,		
Sodium carbonate,		
Hydrochloric acid.		

Dissolve the salt in the water. Slake the lime, mix it with 100 ml of water, and add the mixture to the salt solution.

Heat to the boiling point. Filter. Add a solution of barium chloride to the filtrate as long as it causes precipitation. Then add solution of sodium carbonate until all the calcium and barium shall have been separated. Filter. Neutralize with hydrochloric acid. Evaporate to crystallization or granulation, and dry the salt thoroughly.

Notes. Common salt contains magnesium and calcium salts (chlorides and sulphates). These are precipitated as described.

Description.—Colorless crystals or a white, granular crystalline powder; dry; odorless, and of purely saline taste. Not hygroscopic. Soluble in 2.8 parts of water at 15°, and in 2.5 parts at 100°. Almost insoluble in alcohol. The water-solution is neutral to litmus paper.

SODIUM HYDROXIDE SOLUTION.

[Liquor Sodæ; U. S. P. 1890.]

An aqueous solution containing 5 per cent of sodium hydroxide, $NaOH=40$.

Sodium carbonate	17 parts
Calcium oxide	5 parts
Distilled water, sufficient.	

Dissolve the sodium carbonate in 40 parts of boiling distilled water. Slake the lime and make of it a smooth mixture with 40 parts of distilled water and heat this mixture to boiling. Then add gradually the sodium carbonate solution to the "milk of lime," and boil the whole mixture for ten minutes. Remove the heat, cover the vessel tightly, and, when the contents are cold, add enough distilled water to make the whole weigh 100 parts. Lastly strain the liquid through linen, or, when the precipitate has subsided, draw off the clear solution by means of a syphon.

Reaction. $Na_2CO_3+Ca(OH)_2=2NaOH+CaCO_3$.

Notes. The sodium carbonate employed must, of course, be the crystallized salt, not in any degree effloresced.

The lime might advantageously be washed free from dust and salts before being used. This is done as follows: Slake the 5 parts of lime with about 40 parts of water; then add about 500 parts of water and stir the mixture occasionally during half an hour; let the calcium hydroxide subside, decant the liquid and throw this away. Then add 40 parts of pure water, heat to boiling and use this liquid with the solution of sodium carbonate as described in the formula above given.

A considerable excess of lime is used, but as calcium hydroxide is insoluble in a solution of sodium hydroxide no lime will be contained in the product. An unnecessary large excess of lime is, however, objectionable because the superfluous calcium hydroxide does not subside as readily as the calcium carbonate.

If cold liquids are used the calcium carbonate formed by the reaction is so finely divided and light that it subsides very slowly and the bulky precipitate retains much liquid causing corresponding loss of product.

More concentrated solutions can not be used because the reaction will not then be complete. It is completed when the liquid no longer effervesces with dilute acid or when it does not cause turbidity when dropped into a clear solution of calcium hydroxide.

Solution of sodium hydroxide is usually clarified by subsidence and decantation; but it may, if preferred, be successfully filtered through coarsely powdered and washed marble.

The strength of such a preparation as solution of sodium hydroxide should not be fixed by the quantities and proportions of the materials; it should be tested quantitatively with volumetric solution of sulphuric acid and the strength adjusted by dilution in accordance with the result. In order to carry out this plan the quantity of water used in the process may be diminished ten per cent and the product diluted as required; or the amount of water directed may be used and the product diluted, or concentrated by evaporation, as may be found necessary.

Solution of sodium hydroxide attacks organic matter energetically and is therefore discolored by contact with organic substances. It also attacks ordinary flint glass so that if it be put in glass-stoppered bottles of that kind of glass the stoppers usually become firmly fixed and can not be removed, necessitating the breaking of the bottles to get at the contents. The solution should, therefore, be kept in bottles made of green glass which is not so easily attacked by the alkali, and the stoppers should be coated with a little paraffin or petrolatum.

Description.—The official sodium hydroxide solution is a clear, colorless and odorless liquid, having a very acrid and caustic taste, a corrosive action on organic matter, and a strongly alkaline reaction.

Its specific weight is about 1.059 at 15°C. and the specific volume 0.944.

SODIUM OLEATE.

OLIVE OIL SOAP.

["Medicinal Soap."]

Olive oil	100 parts
Sodium hydroxide	15 parts
Distilled water	35 parts
Alcohol	30 parts

Dissolve the sodium hydroxide in the water, add the oil and the alcohol, and heat the mixture gently, in a porcelain dish, over a water-bath, stirring constantly, until saponification is completed. Then add 300 parts of warm distilled water and mix well.

Make a solution of—

Sodium chloride	25 parts
Sodium carbonate	5 parts
Distilled water	80 parts

and filter it. Add this solution to the solution of the soap, and mix thoroughly.

Heat the mixture over the water-bath, stirring gently, until the soap separates and rises to the surface of the liquid. Let the dish and contents cool. When cold separate the mother liquor from the soap, and rinse the soap with a little cold water. Then squeeze the water out of the soap by means of strong pressure, dry the mass with the aid of gentle heat, and powder the product.

Notes. The quantity of sodium hydroxide prescribed is slightly in excess of the amount theoretically required. All hard soaps consist mainly of sodium oleate. The best Castile soap approaches pure sodium oleate sufficiently for all practical purposes in pharmacy, including the preparation of other oleates by "double decomposition." No soda soap should be used for the preparation of other metallic oleates except after complete drying; hence *powdered* soap is to be used. This is because the amount of moisture in powdered soap can not vary a great deal.

The soap is completely soluble in water although the solution is not clear. Soda soap is precipitated from its water solution by sodium chloride in the manner described.

SODIUM PHOSPHATE.

$Na_2HPO_4.12H_2O=358.$

[Di-Sodium-Hydrogen Phosphate.]

Bone-ash, in fine powder	10 parts
Sulphuric acid	6 parts
Sodium carbonate, sufficient.	

Wash the bone-ash with ten times its weight of boiling water. Decant and reject the water. Mix the wet mass with the acid, set the mixture in a warm place for three days, replacing the water vaporized by the heat, and stir occasionally. Then add 10 parts of boiling water, mix thoroughly, transfer the mixture to a muslin strainer, and let the liquid pass through. Reserve the liquid. Transfer the magma from the muslin to an earthenware vessel, add 10 parts of boiling water, stir well, and again strain the mixture through muslin, adding the colature to that previously reserved. Continue washing the residue with boiling water until the liquid passes nearly tasteless. Mix the several acid liquids and evaporate the mixture to 20 parts. Set this aside a few days. Filter. Heat the filtrate to the boiling point, and gradually add to it a solution of sodium carbonate in hot water, until a filtered portion of the now alkaline liquid effervesces when mixed with dilute sulphuric acid. Digest the mixture an hour or two, and then filter. Wash out the remaining sodium phosphate from the residue by means of hot water, adding the washings to the filtrate. Then evaporate the mixed liquids to 10 parts, and set aside to crystallize.

Purify the product by re-crystallization after dissolving it in twice its weight of water. Repeat the recrystallization as many times as may be necessary to obtain a clean product.

Dry the crystals with as little exposure to the air as possible, to avoid efflorescence.

Reaction. First the tri-calcium phosphate (bone-ash) is decomposed by the sulphuric acid yielding acid calcium phosphate and calcium sulphate as follows:

$$Ca_3(PO_4)_2+2H_2SO_4=CaH_4(PO_4)_2+2CaSO_4.$$

Then the acid phosphate of calcium is decomposed by the sodium carbonate as follows:

$$CaH_4(PO_4)_2+Na_2CO_3=CaHPO_4+Na_2HPO_4+CO_2+H_2O.$$

Notes. When the bone-ash is treated with sulphuric acid the mixture becomes hot. Usually the powdered bone-ash is not washed previous to mixing it with the sulphuric acid, and no water added until after the powder and acid have first been thoroughly mixed. If organic matter be present in the bone-ash, sulphur dioxide is given off. Some carbon dioxide will also be given off from calcium carbonate in the bone-ash. If the bone-ash is washed with water, the object of which is the removal of alkalies, the wash-water should be drained off thoroughly before mixing the moist mass with the acid. When the process is carried out on a small scale the washed and drained bone-ash, still wet, should be put in a tared vessel, the sulphuric acid mixed with it, and finally enough boiling water added to make the whole mixture weigh three times as much as the bone-ash used. After the mass has been digested a sufficient time to insure thorough decomposition of the basic calcium phosphate, to facilitate which the mass should be frequently and thoroughly stirred, the next step is the separation of the liquid containing the acid phosphate of calcium, with which the sodium phosphate is to be made, from the white mass of calcium sulphate which is to be thrown away after the acid liquid has been completely washed out from it and collected.

The acid liquor is boiled down to twice the weight of the bone-ash used and allowed to stand a few days in order that the calcium sulphate held in solution may be deposited. It is then filtered, diluted with an equal volume of water, and heated to the boiling point. To the hot liquid is now added hot sodium carbonate solution as described.

Hot liquids are employed in order that the precipitate formed may be heavy, so as to separate as rapidly as possible. The sodium carbonate must be added gradually, because of the effervescence caused by the escaping carbonic oxide. Litmus paper cannot be used to ascertain when sufficient sodium carbonate has been added, because sodium phosphate itself has an alkaline reaction. After the further addition of sodium carbonate has ceased to cause effervescence, it is necessary to remove a small portion of the liquid, filter it, and test it by pouring it into a small amount of dilute sulphuric acid, when it should effervesce. The slight excess of sodium carbonate added is necessary to insure satisfactory crystallization of the sodium phosphate, and the carbonate remains in the mother liquor.

The crystallization is best effected when an excess of sodium carbonate is present and the solution is not too concentrated. It should not be hastened by evaporating down the solution too far, because the salt then crystallizes with only 7 instead of 12 molecules of water of crystallization.

The product generally requires purification by recrystallization to obtain a sodium phosphate which answers the description and tests of the Pharmacopœia.

Description.—Colorless, transparent crystals, having a specific gravity of 1.55. They effloresce readily, and when slightly heated melt in their water of crystallization.

The salt is odorless, and has a cooling, saline, somewhat alkaline, taste. Efflorescent.

Soluble in 5.8 parts of water at 15°, and in 1.5 parts of boiling water. Insoluble in alcohol. The water-solution is alkaline to litmus paper, but not to phenolphtalein paper.

Recrystallized Sodium Phosphate.

Sodium carbonate	1 part
Commercial sodium phosphate	50 parts
Water	250 parts

Dissolve the sodium phosphate in the water with the aid of heat, filter the solution, put it in a crystallizer and set this aside at perfect rest in a place free from dust that crystals may form by spontaneous evaporation of the solvent at the ordinary room temperature.

Should crystallization be much delayed place a few clear crystals of pure sodium phosphate in the solution to start it.

Collect the crystals in the usual way, drain them, press them gently between cloths or bibulous paper, dry them hastily, and at once put the product in dry bottles, which must be tightly closed and kept in a cool place.

SODIUM PYROPHOSPHATE.

$Na_4P_2O_7.10H_2O=446.$

Prepared by heating sodium phosphate in an iron dish, raising the heat gradually until the salt fuses in its water of crystallization, then falls to powder as that water is expelled, and finally decomposes at a temperatures of 240° to 300° C., being converted into normal sodium pyrophosphate. The dry powder is dissolved in a sufficient quantity of boiling water—about five times its weight—the solution is filtered while hot, and then set aside to crystallize. More crystals are to be obtained from the mother liquor upon evaporation.

Reaction. $2Na_2HPO_4=Na_4P_2O_7+H_2O$.

Notes. Sodium phosphate, in solution, yields a yellow precipitate with test-solution of silver nitrate acidulated with nitric acid; sodium pyrophosphate yields a perfectly white precipitate with the same reagent. The conversion of the phosphate is, therefore, known to be completed when a sample of the dry powder, dissolved in water, yields a white precipitate with silver nitrate.

From 716 parts of sodium phosphate, the yield of pyrophosphate of sodium should be 446 parts, or nearly 62 per cent.

Description.—Colorless, transparent crystals, odorless, of cooling, saline, somewhat alkaline, taste. Soluble in 12 parts of water at 15° and in 1.1 parts of boiling water. Insoluble in alcohol. The water-solution has a slightly alkaline reaction on both litmus paper and phenolphtalein.

SODIUM TARTRATE.

$Na_2C_4H_4O_6.2H_2O=230$.

Tartaric acid	130 parts
Sodium carbonate	251 parts
Distilled water.	

Dissolve the sodium carbonate in the water. Add the tartaric acid gradually, stirring after each addition until dissolved. Filter, evaporate and crystallize, or evaporate the filtrate to dryness to obtain a granular product.

Reaction. $Na_2CO_3+H_2C_4H_4O_6=Na_2C_4H_4O_6+H_2O+CO_2$.

Notes. 148 parts of sodium bicarbonate may be used instead of 251 parts of normal sodium carbonate.

Description.—Colorless, transparent crystals; odorless; of cooling, mildly saline taste. Soluble in 5 parts of water at 15°. Insoluble in alcohol. Neutral to litmus paper

SODIUM THIOSULPHATE.

$Na_2SO_3S.5H_2O=248$.

["Sodium Hyposulphite."]

This salt, which is a monothiosulphate, may be prepared by boiling sulphur with a solution of sodium hydroxide and then passing sulphur dioxide into the liquid until colorless. The solution is then brought to crystallization.

It may also be made by boiling sulphur with a solution of sodium sulphite.

Commercial sodium thiosulphate may be purified by recrystallization. For this purpose it should be dissolved in its own weight of distilled water, the solution filtered, and evaporated slowly. Large crystals are easily obtained.

It should be kept in tightly closed bottles.

Description.—Colorless, odorless crystals, soluble in 0.65 part of water at 15° C. and in about 0.5 part at 20° C. Insoluble in alcohol.

["Sodium hyposulphite" is an unfortunate misnomer for real sodium hyposulphite is Na_2SO_2.]

STRONTIUM CARBONATE.

$SrCO_3=147$.

Dissolve any desired quantity of commercial strontium nitrate in ten times its own weight of distilled water. Add gradually and during constant stirring a water-solution of potassium dichromate to precipitate any barium present and until a clear yellow liquid is produced. Let the liquid stand for 24 hours; then filter. Add to the filtrate, heated at a temperature of 100° C., a sufficient quantity of sulphurous acid to cause the disappearance of the characteristic color of the chromic acid and the appearance of a greenish color due to the formation of chromic salt, meanwhile maintaining the temperature at the boiling point of water. Boil the liquid until the excess of sulphurous acid is expelled. Continue the boiling and add enough strontium carbonate to cause the complete precipitation of the chromium by the strontium carbonate. Filter. Pour the filtrate into a filtered solution of sodium carbonate used in excess. Wash the precipitate until the washings are tasteless and leave no perceptible residue on evaporation. Dry the product at a moderate heat.

Description.—An amorphous, white, odorless and tasteless, insoluble powder.

STRONTIUM CHLORIDE.

$SrCl_2.6H_2O=265.8$.

Strontianite, in powder	1000 Gm
Hydrochloric acid (32% of HCl)	1550 Gm
Strontium hydroxide	25 Gm
Water	500 Gm

Mix the powdered strontianite in a porcelain dish with the water. Heat the mixture to about 60°. Stir well. Add the acid in several portions, waiting after each addition until the effervescence has subsided before adding more. When all of the acid has been added heat to boiling for half an hour. Then add the strontium hydroxide and stir well. The solution should be alkaline in reaction; if it is not, add more of the strontium hydroxide to produce an alkaline reaction. Keep the liquid hot for an hour longer, stirring frequently. Then filter. Add enough hydrochloric acid to the filtrate to render it acid. Evaporate to 1.30 sp. w. Set it aside to cool and crystallize. Drain and dry the crystals.

The mother liquor yields more salt on evaporation in the usual way.

Reaction. $SrCO_3+2HCl=SrCl_2+H_2O+CO_2$.

Notes. The strontium chloride thus obtained is not pure. The principle impurity is barium chloride, for the strontianite contains barium carbonate. Calcium salt is also contained in the product.

It may be partially purified as follows:

Dissolve it in water. Add enough ammonia water to impart an alkaline reaction on litmus paper. Stir well. Filter. Add enough diluted sulphuric acid to the filtrate to render the liquid decidedly acid in reaction. Let stand for two days. Filter. Add enough pure strontium carbonate to neutralize the free acid and leave an excess of strontium carbonate undissolved. Set the mixture in a warm place for a day. Filter again. Evaporate the filtrate to dryness. Dissolve the residue in three times its weight of water, filter, and evaporate to crystallization.

The barium chloride may also be partially precipitated from the solution of strontium chloride by the addition of alcohol in which strontium chloride is soluble while the barium chloride is insoluble in it. Or the strontium chloride may be dissolved in alcohol, the solution filtered and evaporated to dryness, the salt being then recrystallized from a water solution.

Description.—Long, colorless, deliquescent crystals, readily soluble in water and in alcohol.

STRONTIUM NITRATE.

$Sr(NO_3)_2=211.$

Strontianite, in powder	200 parts
Nitric acid (68% of HNO_3)	250 parts
Strontium hydroxide	5 parts
Water	300 parts

Mix the strontianite with the water. Warm the creamy mixture. Add the nitric acid gradually, stirring well and taking care not to allow the liquid to run over from a too violent effervescence. When all of the acid has been added, heat the liquid to expel the carbon dioxide. Add the strontium hydroxide, stir well, and digest for an hour, stirring frequently.

Filter. Acidify the filtrate with nitric acid. Evaporate nearly to dryness, and place the wet crystalline mass of salt in a funnel to drain. Dry the product with the aid of moderate heat.

Reaction. $SrCO_3+2HNO_3=Sr(NO_3)_2+H_2O+CO_2$.

Description.—Transparent, anhydrous crystals, readily soluble in water.

ZINC ACETATE.

$Zn(C_2H_3O_2)_2.2H_2O=219$.

Zinc oxide	300 Gm
Acetic acid	1000 ml
Distilled water	1000 ml

Mix 600 ml of the acid with the whole of the water in a flask, add the zinc oxide, agitate gently, and warm the mixture to about 60° C.; add the remainder of the acid gradually, continuing the digestion until no more of the oxide is dissolved. Heat the solution to the boiling point; filter while hot, add a little acetic acid (about 10 ml) to insure that the acid is present in slight excess. Set the solution in a cool place for two days to crystallize. Decant the mother liquor, evaporate it over a water-bath to one-half, acidulate with acetic acid as before, if necessary, and again set aside to crystallize in a cool place. Put the crystals in a funnel to drain; then dry them on filter paper or on a porous tile, without the aid of heat.

Must be kept in a tightly-corked bottle.

Reaction. $ZnO+2HC_2H_3O_2=Zn(C_2H_3O_2)_2+H_2O$.

Notes. The same result will be obtained if 420 Gm of zinc carbonate to be used instead of 300 Gm of the oxide. The carbonate should be added gradually on account of the effervescence caused by the liberation of carbonic acid, and the solution formed is then to be boiled a few minutes to expel the carbonic acid perfectly.

When the preparation is made on a manufacturing scale from commercial zinc oxide or carbonate, it is necessary to digest the solution of acetate of zinc for several days with some pure metallic zinc, or with pure zinc oxide, in order to remove any lead, copper, cadmium and iron that may be present. By this digestion, however, some basic zinc acetate is formed, which must be converted into normal acetate by the addition of more acetic acid before crystallizing. A solution of basic salt will not crystallize, but the presence of a little free acetic acid facilitates the crystallization.

When the first crop of crystals has been collected, the mother liquor must be evaporated at a temperature not above 80° C. (176° F.) until a pellicle forms, after which it is set aside in a cool place. If the evaporation is carried too far a precipitate of zinc acetate containing but one molecule of water of crystallization will deposit.

Zinc acetate may also be made by double decomposition from 38 parts of lead acetate and 29 parts of zinc sulphate, each dissolved in 80 parts of distilled water, the solutions being filtered and then mixed. The reaction is then:

$$Pb(C_2H_3O_2)_2+ZnSO_4=Zn(C_2H_3O_2)_2+PbSO_4.$$

Description.—Soft, white, pearly crystals, of a faintly acetous odor and an astringent, afterwards metallic taste. Soluble in 2.7 parts of water and in 36 parts of alcohol at 15°; in 1.5 parts of boiling water, and in 3 parts of boiling alcohol. Reaction acid.

ZINC BROMIDE.

$ZnBr_2=223.$

Zinc sulphate	8 parts
Potassium bromide	7 parts
Boiling distilled water, sufficient.	

Dissolve the sulphate in 10 parts and the bromide in 6 parts of the water. Mix the solutions while hot. When cool add 40 parts of alcohol, and filter the mixture through asbestos. Evaporate the filtrate to dryness, and granulate the product.

Reaction. $ZnSO_4+2KBr=K_2SO_4+ZnBr_2.$

Zinc bromide may also be made from zinc sulphate and barium bromide.

Another Method.

Zinc	66 parts
Bromine	160 parts
Water	250 parts

Put the zinc and water in a flask of about 500 Cc. capacity. Add the bromine in small portions at a time, waiting after each addition until the red color has nearly disappeared and the reaction subsided. When all of the bromine has been added, filter the liquid and evaporate to dryness.

Description.—A white, granular salt; odorless; of sharp, saline, metallic taste. Very hygroscopic. Readily soluble in water and in alcohol.

ZINC CARBONATE; PRECIPITATED.

$2ZnCO_3.3Zn(OH)_2$=547.

Zinc sulphate	20 parts
Sodium carbonate	21 parts
Boiling water, sufficient.	

Dissolve the sodium carbonate in 40 parts of the water in a porcelain capsule. Dissolve the zinc sulphate in an equal quantity of boiling water. Place the capsule containing the solution of sodium carbonate on a sand-bath so as to keep the solution hot; then gradually add the solution of zinc sulphate, stirring briskly. Boil the mixture for fifteen minutes after the effervescence has ceased; then let the precipitate subside. Decant the mother-liquor and throw it away. Add 120 parts of boiling water to the precipitate, and stir well, let settle once more, and repeat the washing by affusion and decantation of hot distilled water until the washings cease to give any precipitate with test solution of barium chloride. Collect the precipitate on a muslin strainer or on a paper filter, let it drain thoroughly, and then dry it at a gentle heat.

Reaction.

$$5Na_2CO_3+5ZnSO_4+3H_2O$$
$$=2ZnCO_3.3Zn(OH)_2+5Na_2SO_4+3CO_2.$$

Notes. The solutions are used hot in order to expel all the carbonic acid which is set free, carbon dioxide escaping with effervescence. If cold solutions are used a portion of the precipitate redissolves in the carbonic acid held in the liquid. The zinc sulphate is to be added to the sodium carbonate in order to keep the latter in excess, and by having the soda solution hot, the further advantage is gained that the precipitate is more readily washed free from sodium salt.

Description.—A fine, white, odorless and tasteless, insoluble powder.

ZINC CHLORIDE.

$ZnCl_2=135.8.$

A solution of zinc chloride is prepared as described in the article below. It is then evaporated to dryness and the residue fused, or the product is granulated. It must be bottled while warm in warm dry bottles, and should be kept in small, tightly-stoppered bottles.

Description.—Zinc chloride should be perfectly white. It is very deliquescent. Its solution is strongly acrid, corrosive, and of acid reaction.

Zinc Chloride Solution.

The official solution of zinc chloride (U. S. P., 1890) contains about 50 per cent of $ZnCl_2$. It is prepared as follows:

Granulated zinc	240 Gm
Hydrochloric acid	840 Gm
Nitric acid	12 Gm
Precipitated zinc carbonate	12 Gm
Distilled water, sufficient.	

Put the zinc in a glass or porcelain vessel, and add 150 ml of distilled water. Then add the hydrochloric acid gradually. Digest until the acid shall have become saturated. Decant the solution from the undissolved excess of zinc.

Add the nitric acid to the solution, evaporate the mixture to dryness and heat the dry mass to fusion at a temperature not exceeding 115° C.

Let the fused zinc chloride cool and then dissolve it in a sufficient quantity of distilled water to make the weight of the solution one thousand grams.

Add the precipitated zinc carbonate, agitate the mixture occasionally during twenty-four hours, and then set it aside, at rest, until clarified by subsidence.

Finally separate the clear solution by decantation or by means of a glass syphon, and keep it in glass-stoppered bottles.

Reaction. $Zn + 2HCl = ZnCl_2 + H_2$.

Notes. The metal dissolves in the hydrochloric acid with the liberation of heat and the evolution of hydrogen. Instead of adding the acid to the zinc, the metal may be added to the acid.

The effervescence caused by the escape of the hydrogen may be very active at first, but subsides as the zinc chloride increases and the acid diminishes. When but little acid remains digestion is necessary to cause complete saturation, and further digestion is desirable to cause the separation of certain foreign metals which may have been contained in the zinc.

Commercial zinc is usually contaminated with one or more of the metals, copper, lead, arsenic, cadmium, iron and manganese. When an excess of zinc is used, so that a portion of it remains undissolved at the end of the digestion, several of these other metals dissolved together with the zinc are precipitated by the latter on prolonged digestion.

But one of the most common impurities is iron, which can not be precipitated by digestion with an excess of zinc. To remove the iron and (manganese) nitric acid is added to convert the ferrous chloride to ferric, and the solution is evaporated to dryness and the residue fused to expel the excess of nitric acid. The fused zinc chloride (containing any iron chloride derived from the iron in the zinc used) is then redissolved and the solution shaken with precipitated zinc carbonate, which removes the iron which is precipitated in the form of hydroxide.

Solution of zinc chloride cannot be filtered through paper, because the latter swells until its pores are closed and is then corroded. It may be filtered through powdered and washed glass, or through purified asbestos. Decantation is sometimes sufficient. A dilute solution may be filtered through paper, but the organic substances then taken up by it blacken the product as soon as sufficiently concentrated by evaporation.

Description.—A clear, colorless, odorless liquid of astringent, sweetish, metallic taste and acid reaction. Corrosive in its action upon organic matter. Sp. w. about 1.535 at 15°.

ZINC OLEATE.

$Zn(C_{18}H_{33}O_2)_2 = 627.$

Zinc acetate	2 parts
Powdered white castile soap	5 parts

Dissolve the zinc acetate in 300 parts of water, and the soap in 200 parts of hot water. Add the cold soap solution to the solution of zinc acetate, stirring briskly. Drain the precipitated zinc oleate on a muslin strainer, and wash it several times with cold water. Dry it on paper or muslin, without the aid of heat.

Reaction. $2NaC_{18}H_{33}O_2 + Zn(C_2H_3O_2)_2 = Zn(C_{18}H_{33}O_2)_2 + 2NaC_2H_3O_2.$

Notes. The sodium acetate is easily washed away from the precipitated oleate. The yield is about 2.4 parts.

Description.—A soft, white powder, having a soapy feel.

ZINC OXIDE.

$ZnO=81$.

Calcine the official zinc carbonate in a dish at a low red heat until a cooled sample, mixed with a little water, no longer effervesces with diluted hydrochloric acid.

Reaction. $2(ZnCO_3).3Zn(OH)_2=5ZnO+3H_2O+2CO_2$.

Notes. Too high heat renders the product yellow, and this yellowish tint may be retained for a long time. [All pure zinc oxide turns yellow when heated but becomes white again on cooling unless it was heated too strongly. A zinc oxide containing much iron is always discolored even if not overheated in the process of calcination.] Overheating also makes zinc oxide less soft to the feel.

ZINC PHENOLSULPHONATE.

$Zn(C_6H_5SO_4)_2.8H_2O=555$.

(Sulphocarbolate of Zinc.)

Crystallized phenol	20 parts
Sulphuric acid	25 parts
Barium carbonate	40 parts
Zinc sulphate	29 parts
Water.	

Add the phenol to the acid, mix well, and heat the mixture at 55° in a porcelain dish over a water-bath for several days. Add 400 parts of water and mix well. Then neutralize by adding the barium carbonate, stirring the whole thoroughly. Filter. Reject the precipitate. To filtrate add the zinc sulphate previously dissolved in 200 parts of water. Now filter away the precipitated barium sulphate, slightly acidulate the filtrate with sulphuric acid, and evaporate to crystallization.

Purify the product by repeated re-crystallizations.

Reactions. $2HC_6H_5SO_4+BaCO_3=Ba(C_6H_5SO_4)_2+H_2O+CO_2$; and then, $Ba(C_6H_5SO_4)_2+ZnSO_4=Zn(C_6H_5SO_4)_2+BaSO_4$.

Notes. The solution of zinc sulphate should be added to the solution of sulpho-carbonate of barium until exactly precipitated—that is, the further addition of zinc sulphate must be discontinued as soon as it no longer causes any further precipitation. Sulphocarbolate of zinc crystallizes more readily, and without reddish color, from a solution acidulated with sulphuric acid.

Description.—Colorless crystals, readily soluble in water and in alcohol.

ZINC SULPHATE.

$ZnH_2SO_5.6H_2O=287$.

Zinc clippings, or granulated zinc......	4 parts
Sulphuric acid	5 parts

Dilute the acid with 30 parts of water; add the zinc gradually. When effervescence has ceased, digest with the undissolved portion of the metal for a few days. Filter. Evaporate the filtrate until a drop becomes turbid on cooling. Then let the solution become cold, stirring constantly so that small crystals may be formed. Collect and dry these on muslin. Evaporate the mother liquor to obtain more crystals.

Reaction. $Zn+H_2SO_4+7H_2O=ZnH_2SO_5.6H_2O+H_2$.

Notes. The zinc should be nearly free from other metals. If it contains iron, the zinc sulphate contaminated with ferrous sulphate will become yellow in time by the oxidation of the ferrous to ferric salt. Zinc sulphate containing iron will, of course, be unfit for the preparation of zinc oxide.

Any iron and manganese present in the metal will not be removed by digestion with an excess of zinc; but lead, copper, and cadmium, if present, are thus separated. The gray flocculi which are formed when the zinc is dissolved in the acid are mainly lead.

Pure zinc dissolves with difficulty in the acid; if it contains iron it dissolves more rapidly in proportion to the iron in it.

Iron is removed as described below in the recrystallization of zinc sulphate.

The solution should not be too concentrated for crystallizing, for when the salt is crystallized from a warm solution (over 30° C.) the crystals formed contain less water of crystallization ($ZnSO_4.5H_2O$). The crystals should be dried without the aid of heat, or at least at a temperature not exceeding 30° C.

Impure zinc sulphate may be purified to a great extent by recrystallization. The salt crystallizes best from a slightly acid solution.

Zinc sulphate may also be made by saturating sulphuric acid with zinc oxide and crystallizing.

If large crystals are desired let the saturated solution be evaporated slowly at the ordinary temperature and without disturbing it.

Description.—Colorless, transparent crystals; odorless; taste metallic, astringent. Soluble in 0.6 part of water at 15°, and in 0.2 part of boiling water; also in 3 parts of glycerin. Insoluble in alcohol. Reaction acid.

Recrystallized Zinc Sulphate.

White vitriol........................	5 parts
Water	3 parts

Make a solution. Acidify it with 1 part of diluted sulphuric acid. Boil it for a few minutes. Filter. Conduct a current of chlorine into the warm solution until it acquires the odor of it. Add a small quantity of iron-free zinc oxide or zinc carbonate to produce a slightly turbid mixture containing but little undissolved zinc compound. Digest for a day or two, shaking occasionally. Bring it to the boiling point. Filter. Acidify again with diluted sulphuric acid. Crystallize in the usual way.

Notes. White vitriol contains iron, and consists of an opaque, granular, more or less dirty salt mass. When its solution is strongly acidified and boiled it can be filtered quite clear. Chlorine converts the iron present into ferric sulphate. The iron is then easily precipitated by digestion with zinc oxide or zinc carbonate in slight excess. The separation of the iron is facilitated by boiling. After filtration the solution is again to be acidified by the addition of about 1 part of diluted sulphuric acid because the salt crystalizes most satisfactorily from an acid solution.

If copper, lead, arsenic, cadmium, or manganese be present, these metals are removed as well as iron by this method.

ZINC SULPHITE.

$ZnSO_3.2H_2O=181$.

Zinc sulphate	60 parts
Sodium sulphite	53 parts
Distilled water, sufficient.	

Dissolve each salt separately in 200 parts of cold distilled water and filter the solutions. Add the zinc salt solution to the solution of the sodium sulphite, stirring well. A precipitate of zinc sulphite will be formed in the course of from twenty to thirty minutes.

Decant the mother-liquor. Wash the precipitate with a limited amount of cold water until the washings are nearly tasteless and no longer contain sodium sulphate. Dry the product without the aid of heat.

Reaction. $ZnSO_4+Na_2SO_3=ZnSO_3+Na_2SO_4$.

Notes. Cold solutions are used and the product washed and dried at the ordinary room temperature because zinc sulphite is liable to be decomposed at a higher temperature, basic salt being formed.

Description.—A white, crystalline powder, practically insoluble in water.

ALOIN.

50 Gm of aloes are mixed with 300 Cc of hot water and stirred until the particles of aloes are disintegrated; then a few drops of hydrochloric acid are added. On cooling, the

solution is decanted from the separated resin and there is added to it rapidly 50 Cc of ammonia water (20%) and then a solution of 15 Gm of calcium chloride in 30 Cc of water. The aloin-calcium compound separates out and after fifteen minutes is collected on a strainer, drained and strongly expressed. It is then decomposed by trituration in a mortar with a slight excess of hydrochloric acid. The mixture of aloin and calcium chloride which results is dissolved in a small amount of boiling water, filtered and the filter washed with a little hot water. The aloin is crystallized out of the solution by setting it aside at a low temperature. The crystals are collected and dried.

Description.—A yellow micro-crystalline powder, with a slight odor of aloes and a very bitter taste.

AMYGDALIN.

$C_{20}H_{27}NO_{11}=457$.

Bitter almonds, ground..................	100 Gm

The almond meal is shaken in a strong bottle with about 300 Cc of Benzin for 15 minutes. The mixture is then strained and the residue strongly expressed. The residue is again treated as before with another portion of Benzin.

The dried residue together with about 200 Cc of alcohol is placed in a flask connected with a reflux condenser and boiled gently for twenty minutes. The mixture is strained while hot, and the residue expressed. The same treatment is repeated on the residue with a second portion of alcohol. The strained liquids are united, heated, and filtered while hot. The alcohol is then distilled from the filtrate until only a small volume remains. This is mixed with one half its volume of ether, and set aside until the amygdalin deposits. The separated crystals are washed with a little ether and recrystallized from about 100 Cc of boiling alcohol.

Description.—White pearly scales, odorless and slightly bitter. Readily soluble in water and in 900 parts of cold or 11 parts of boiling alcohol. Crystals deposited from water are prisms with three molecules of water of crystallization.

BERBERINE HYDROCHLORIDE.

$C_{20}H_{17}NO_4HCl+4H_2O=443.4$.

Barberry bark, No. 40 powder........	100 Gm

Moisten the powder with a mixture of 60 Cc of water and 5 Cc of acetic acid and macerate for twelve hours. Then percolate the drug to exhaustion with water and evaporate the percolate to about 60 Cc. Add 5 Cc of hydrochloric acid and set aside for twenty-four hours. Collect the crystals on a wetted strainer and wash with water. Dry the crystals, dissolve them in fifty times their weight of boiling water and filter. To the filtrate when cooled to 40° C. add one half its volume of acetone, make strongly alkaline with solution of sodium hydroxide and set aside for about 12 hours. Collect the berberine-acetone compound on a filter, wash and dry it. Then add to it enough diluted hydrochloric acid to neutralize the berberine, heat over a water-bath until the acetone is driven off and set aside for twenty-four hours. Collect the crystals, wash with a little water and dry them.

Note. Other salts of berberine may be prepared by decomposing the berberine-acetone with the appropriate acid.

Description.—Small crystalline needles of a golden yellow color. Sparingly soluble in water or alcohol at the ordinary temperature.

OIL OF CLOVES.

Cloves, coarsely ground	35 Gm
Sodium chloride	20 Gm
Water	230 Cc

Mix in a 500 Cc flask and allow it to stand 2 hours. Then distill over a sand-bath until 100 Cc of distillate are obtained. Agitate the distillate and allow it to stand until the oil separates from the water. Decant off the supernatant layer, return it to the flask and redistill as before. Repeat the operation until the cloves have been deprived of the oil. Finally collect the oil.

Description.—A colorless or pale yellow, thin liquid having the aromatic odor of cloves and a pungent spicy taste. Specific gravity 1.040 to 1.060 at 25° C.

PIPERINE.

$C_{17}H_{19}NO_3=285.$

White pepper, in No. 30 powder......	50 Gm

Exhaust the powdered pepper by percolation with strong alcohol. Distill off the alcohol by means of a water-bath until a soft extract remains.

Stir the extract thoroughly with about 25 Cc of potassium hydroxide solution and decant the liquid from the residue. Repeat the washing, with several portions of potassium hydroxide solution to remove resinous matter. Dry the residue well, dissolve it in hot alcohol, filter, and set the filtrate aside to crystallize. Recrystallize.

Description.—Colorless or pale yellow, prismatic crystals without odor. Insoluble in water; soluble in 15 parts of alcohol.

SALICYLIC ACID.

$HC_7H_5O_3=138.$

Natural methyl salicylate...............	5 Gm
Potassium hydroxide..................	4 Gm

The oil is introduced into a flask and the Potassium Hydroxide, previously dissolved in 35 Cc of distilled water, is added. The flask is connected with a reflux condenser and its contents heated by means of a sand-bath until the oil has disappeared. Dissolve the contents of the flask in about 150 Cc of water and then add a slight excess of hydrochloric acid.

The precipitate is collected on a filter and washed with distilled water. Dissolve the precipitate in 150 Cc of boiling water and set aside to crystallize. Recrystallize if necessary.

Reaction. $CH_3C_7H_5O_3+KOH=$
$CH_3OH+KC_7H_5O_3$ and then

$KC_7H_5O_3+HCl=KCl+HC_7H_5O_3$.

Description.—Fine white needles sparingly soluble in cold water. Very soluble in alcohol and ether.

SANTONIN.

$C_{15}H_{18}O_3=246$.

Santonica	250 Gm
Lime	82 Gm
Water	2500 Gm

Boil together one hour, replacing the water lost by evaporation, and strain. Boil the residue with 2,000 Cc of water and 35 Gm of lime for one-half hour and strain. The united liquids are permitted to stand 24 hours in order that any solid particles may subside. Decant the liquid from the sediment and add to it a solution of sodium carbonate as long as a precipitate is formed. Filter, and evaporate the filtrate to 750 Cc. While it is still warm, add enough hydrochloric acid to render it strongly acid. Set aside four or five days and then carefully decant the mother liquor from the crystals. Collect the crystals on a filter and wash them with water until the washings are colorless and neutral, then with a mixture of 10 Cc of ammonia water and 100 Cc of water and afterward with cold water until the washings are colorless. Dry the precipitate and digest it in a flask with animal charcoal and 100 Cc of alcohol for one-half hour at a temperature below 75° C. Filter and set the filtrate aside to crystallize.

Description.—Shining, flattened, rhombic prisms which are colorless and permanent in the air, but turn yellow on exposure to light.

TABLE OF ATOMIC WEIGHTS.

Aluminum	27.
Antimony	119.
Arsenic	75.
Barium	137.
Bismuth	207.
Boron	11.
Bromine	79.
Cadmium	112.
Calcium	40.
Carbon	12.
Cerium	139.
Chlorine	35.4
Chromium	52.
Cobalt	59.
Copper	63.5
Fluorine	19.
Gold	196.
Hydrogen	1.
Iodine	126.
Iron	56.
Lead	205.5
Lithium	7.
Magnesium	24.2
Manganese	55.
Mercury	198.5
Nickel	58.5
Nitrogen	14.
Oxygen	16.
Phosphorus	31.
Platinum	193.
Potassium	39.
Silicon	28.3
Silver	107.
Sodium	23.
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Sulphur	32.
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Tungsten	183.
Zinc	.65

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  sulphite, 99.
  sulphocarbolate, 96.

Φ· 94 94 g 47·5 g
S 98 53 cc 26·5 cc
As 85 g

7.2
5
1.7

98 (84)

142
71
1
14
71

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